PRACTICAL TESTING

OF

RAW MATERIALS.

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MANCHESTER.

PRACTICAL TESTING

OF

RAW MATERIALS.

A CONCISE HANDBOOK FOR MANUFACTURERS, MERCHANTS, AND USERS OF
CHEMICALS, OILS, FUELS, GAS RESIDUALS AND BYE-PRODUCTS,
AND PAPER MAKING MATERIALS, WITH CHAPTERS
ON WATER ANALYSIS AND THE TESTING
OF TRADE EFFLUENTS.

BY

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TABLE OF CONTENTS.

PAGE

CHAPTER I.—THE NECESSITY FOR THE TESTING OF RAW MATERIALS - - - - - - - 1

CHAPTER II.—THE LABORATORY - - - - - - 7

CHAPTER III.—THE TESTING OF FUELS - - - - - - 42

CHAPTER IV.—MISCELLANEOUS CHEMICALS - - - - 51

CHAPTER V.—OIL TESTING - - - - - - - 67

CHAPTER VI.—GAS RESIDUALS AND BYE-PRODUCTS - - 82

CHAPTER VII.—CHEMICALS AND RAW MATERIALS USED IN PAPER MAKING- - - - - - - 93

CHAPTER VIII.—WATER ANALYSIS, PURIFICATION, FILTRATION, AND SOFTENING - - - 117

CHAPTER IX.—TRADE EFFLUENTS - - - - - - - 135

CHAPTER X.—MISCELLANEOUS TESTING, TABLES, &C. - 143

INDEX - - - - - - - - - - - - 165

CHAPTER I.

The Necessity for the Testing of Raw Materials.

THE publication of this manual of practical testing is not an attempt to offer a substitute for the many valuable standard works on chemical analysis which are already in existence The needs of the analytical chemist are well supplied He has access to a whole library of well-written and well-authenticated text books on every department of chemical knowledge, and it would be mere presumption to offer any substitute for the recognised standard works of reference

There exists, however, a want which this volume is designed to meet The general users and merchants of chemicals and drysalteries are not, as a rule, versed in chemical analysis, nor are the exigencies of their business such as to demand the inclusion of a trained chemist among the members of their staff, but there are many occasions when, in buying articles of a chemical nature, it would often be very helpful—and very profitable—to be able to form an opinion as to the relative values of, say, half a dozen samples of the same class of material For instance, a certain large manufacturer had for many years been purchasing the lubricating oil used in his very extensive works upon the guidance of a rough and ready test—he filled a jar with the oil, and ascertained the specific gravity by means of an antiquated hydrometer The denser the oil, the higher it rose in his estimation, and he placed his contracts accordingly But, as his business developed, and he began to use a different class of raw material, he found it necessary to engage a chemist to control the operations of his works, and, in due course, the lubricants came under examination. The chemist was a smart man, and he quickly proved, to the entire satisfaction of his employer, that the bill for lubricants was far higher than was necessary. The manufacturer was amazed to find that there *was* any other test for lubricating oil than the specific gravity test, but, as the time for placing contracts again came round, he obtained a dozen quotations and as many samples These samples were submitted to a very simple test—which will be found set forth in its proper place in these pages—and the result was that the following year's contract was placed at such a figure that the saving effected paid the chemist's salary several times over Of course, this was a large establishment, but the same state of things exists in many works

Another case in point A calico printer in the Manchester district bought bleaching liquor on the test of the hydrometer. He had been purchasing liquor at a certain number of degrees Twaddell, when an enterprising traveller called upon him with a sample which actually tested twice as high in degrees, and at the same price he had been previously paying The bleacher tested the liquor with his own Twaddell glass, but the instrument spoke for itself—it was twice as high in degrees, hence it must be twice as strong in bleaching power. The manufacturer gave a large order for the liquor, and the traveller went on his way rejoicing. The wonderful bleach liquor arrived in due course, but it speedily became apparent that it was doing less work than the *weaker* article, and, after vainly endeavouring to account for this very unsatisfactory result, the manufacturer consulted an analyst, who speedily found that the extra "strength" was attained by the copious addition of common salt, while the "available chlorine"—the active bleaching agent—was even less than in the ordinary liquor which had been discarded

A further example of the necessity for the control of raw materials, and especially of chemicals, may be quoted It is by no means an uncommon case, and also relates to bleaching materials. In the paper trade large quantities of bleaching powder are used, and in some mills no test is ever made, The powder is sold as containing 35 per cent to 35½ per cent of available chlorine, and in many mills the invoice is "taken as read!" A certain paper maker had been receiving his supplies of bleaching powder from one of the largest firms of makers for a number of years, and was very fond of boasting of the excellent qualities of his bleach—it was above suspicion (mainly because it bore a well-known brand). He, too, swore by the Twaddell glass, and it was a difficult matter to persuade him of the necessity for any [illegible] chemical test Eventually, however, a member of the staff, who was possessed of a certain amount of chemical knowledge, obtained permission to exercise his skill upon the supplies of bleach which came into the establishment The results of his work showed that, instead of receiving bleaching powder containing 35½ per cent of available chlorine, the chlorine contents were never more than 33 per cent or 34 per cent When it is remembered that each 1 per cent of chlorine means 3 per cent on the invoice, the meaning of this statement will be appreciated How long this state of affairs had been existing it would be hard to say, but close supervision for five or six weeks showed that no bleach came into this particular mill which tested anything like the contract strength When matters were ripe the attention of the manufacturers was called to the subject, and they at once went into a fit of righteous indignation at the mere suspicion of such a thing as *their* bleach being weak. Such an assumption had never been made before, they protested, and the users were surely mistaken But the paper maker was obdurate, he sustained his claim, and—this is the point—the same makers now send him bleaching powder which *never tests less than 37 per cent of available chlorine!* The apparatus necessary for the test—which is a very simple one—did not cost many shillings, but the effect on the bleach account of the works was such that now the paper maker in question will never admit a delivery of bleach which has not passed the analysis of the works chemist.

Again, with regard to fuel Another manufacturer, who has profited by ripe experience, will not place his coal contracts until he has had the samples submitted to him properly tested for their fuel value. He generally submits to analysis perhaps half a dozen samples of slack from different mines, and the result is that often the highest-priced slack is far from being the most valuable as a fuel, while a sample at a lower figure has a very much higher value for steam-raising purposes. The point of this instance is that this particular manufacturer burns some 5,000 tons of coal yearly, and it does not need much discernment to see that even a small percentage of saving will effect a remarkable difference in a year's coal bill, and with fuel at present prices it is surely worth the while of large coal users [illegible]

Such instances as the foregoing might be multiplied indefinitely, but one more must suffice Again referring to the paper trade, most people are aware that enormous quantities of wood pulp are now used in the manufacture of paper This pulp is sold on a basis of so much per cent of dry fibre in the material—a "dry" pulp must not contain more than 10 per cent of water, and a "wet" pulp must not test more than 55 per cent of moisture In one of the largest mills in England, many thousands of tons of pulp were consumed annually, yet, until comparatively recently, not a bale was ever tested. The manager declared that he dealt only with "honest men," and scouted the idea that he was ever "done" Yet here again, one of the staff—not a trained chemist—found, almost by accident, on the application of some little knowledge of commercial testing, that his firm, although they dealt invariably with "honest men," were paying yearly for hundreds of tons of water at from £3 to £10 per ton. A close system of testing was instituted, and the result is that at the present time this particular firm is effecting a clear saving of close upon £2,000 a year

Wherever chemicals, oils, drysalteries, fuels, or any similar raw materials of varying strength or composition are employed, the quality of the article should *never* be accepted on the word of the seller Instances have been mentioned with a view to illustrating this contention, but the principle should be applied *invariably* wherever goods of uncertain strength are concerned It is well known that many chemical works have two classes of customers—the people who test their goods, and the people who do not test at all The former class gets high strength deliveries, the latter gets whatever is left. Users of chemicals, lubricants, fuels, etc, have very little idea as to the truth of this statement. It is only when they come to apply such simple, yet reliable tests as will be found in these pages, that the situation is seen in its true light. We have nowadays continually dinned into our ears the never-ending story of German superiority in commercial matters, yet one very important factor of German commercial keenness is often lost sight of, and it is this. The German manufacturer does not take the quality of his raw materials for granted, *the English manufacturer very often does.*

It is not necessary that every works should number a trained analyst amongst the members of the staff. There are, comparatively speaking, few works, with the exception of very large ones, which could find work for a trained chemist, and there are fewer still where the willingness to pay the necessary salary would be found! In small works of every description—soap works, paper mills, bleach works, dye works, hat works, small engineering works, textile factories—in all these, and many other establishments, articles which are extremely variable in strength are constantly used in large quantity, yet in very many cases there is no system of testing in operation The reason given is, "We have not enough work for a chemist, and nobody in the place knows how to test the stuff If we send all our samples to a public analyst his fees are so high that it would be ruinous. What are we to do?"

The problem has been solved in some cases, and in a very simple manner The Lancashire paper trade supplies several instances of this A paper maker has realised the necessity for exercising some measure of chemical control over his raw materials; but he is not prepared to meet the expense of submitting every sample to a public analyst, considering that a rough test, in the first instance, is all that is required His attention has been drawn to the fact that some studious member of his clerical staff possesses a little knowledge of chemistry, and, acting upon what has proved a good idea, the manufacturer has encouraged the expansion of this knowledge A few sessions at a technical school, backed up with good text-books, has proved sufficient preparation for the embryo works chemist to enable him to undertake the testing of the more important raw materials, and practice, with the run of the works, has, in several notable cases, borne ample fruit in a very marked degree of economy effected by these simple means.

It is well to add that in these cases an important lesson has been learnt, and it is this.—The testing of raw materials and supplies generally is carried out by the works chemist, who reports any cases where goods are not up to contract strength or quality, but the manufacturer, after a little experience, does not attempt to fight claims for allowances upon the basis of the works test What he does, after a confirmation of the test, is to call in a public analyst, armed with *his* certificate, he proceeds to fight the claim, and it may be stated as a positive fact that in nine cases out of ten the manufacturer wins the day, and secures his allowance—in many cases a very substantial one. This method of procedure is recommended with confidence to those who are desirous of establishing chemical control over their raw materials with a minimum of expense

The object of this volume is to place within easy reach of every user of chemicals, etc, the knowledge which will enable him to control the quality and strength of the materials in question In many cases, the methods of testing given in this manual are not easily accessible to the ordinary user who has not received a chemical training Many of the processes may be found in text-books, but they are frequently buried under a mass of (to the untrained seeker) irrelevant matter, and the instructions for the carrying out of the analytical operations are couched in language which to the uninitiated is sometimes difficult to understand Other methods for the rapid testing of commercial articles are not easily to be found in any of the ordinary text-books, or, if it were possible to find them, it is not always convenient for the manufacturer, merchant, or user to have in his works a library of analytical literature A handy collection of *reliable* tests of the commonly used chemicals, lubricants, fuels, etc, which are consumed in such immense quantities, is a *desideratum*, and in this volume such a collection will be found

CHAPTER II.

The Laboratory.

THE FITTING UP OF THE LABORATORY.

1.—It is very desirable in selecting a room to be converted into a laboratory that several most important points should be kept in mind In the first place there should be ample room for all the operations which are likely to be performed Works laboratories are, generally speaking, at a great disadvantage in that the room set apart for analytical operations is far too restricted No apartment is considered too small to serve for a laboratory In many cases it almost appears as though the chief consideration had been to apportion the smallest room in the whole establishment to the work of testing It is altogether a mistake to stint the laboratory in the matter of space. Select a fairly large room, which will give ample space for comfortable working Every square foot of room in a works is valuable, but no better investment can be made than the apportionment to the laboratory of as large a room as possible, and the larger the better.

The next point is that the laboratory should be easy of access. In some mills the sanctum of the chemist is cunningly hidden away in an almost inaccessible corner of the works, and it would be safe to challenge anyone, not thoroughly familiar with the ins and outs of the works, to penetrate to the repository of what little apparatus the establishment possessed This is a great mistake, for, where the laboratory is hidden away in such a manner, its usefulness is diminished, and especially is this the case when no chemist is numbered amongst the members of the staff, and someone with a smattering of chemical knowledge is supposed to do the testing—such as it is—in his odd moments. The laboratory should be situated in a convenient part of the works—in many new mills it is placed in the same block of buildings as the offices—and, when these are in close proximity to the works proper, it is, no doubt, the best arrangement If a room is fitted up *in* the mill a certain amount of vibration will be inevitable, and this vibration will seriously impair the accuracy of balance work Freedom from vibration, therefore, should be secured

2. Gas and Water—There should be a good service of gas and water If this does not already exist, it should be provided before any serious work is attempted The room should also be well lighted If gas is not available for heating apparatus, spirit lamps must be substituted, but gas is preferable in every respect Having selected a suitable room, and assuming that the essential points of (1) ample space, (2) convenience of access, and (3) gas and water supply have all been met, we will now consider the arrangement of the laboratory.

3. Shelves.—If the room is square, with windows on two sides, then the two sides not pierced with windows should be fitted up with shelving Three shelves, each running along the whole length of the wall, and of a good width, will be found useful for re-agent bottles With regard to the other two walls, the space of which is broken up by the windows, short lengths of shelving may be fitted *between the windows*, for shelf room is extremely valuable in a laboratory, and it should be arranged so that solid reagents are allotted to one part, and liquid reagents to another, while bottles containing samples may be kept entirely separate in a third division. This arrangement will avoid confusion, and, where many samples are tested in the course of a few months, a liberal allowance of shelf room will be appreciated

4. Benches.—The following arrangement is a very convenient one Again assuming that the laboratory is square in shape, benches should be fitted round three sides of the room; in some laboratories all four sides of the room are fitted up with benches, and, if possible, this is all the better for convenience in working A convenient height for a working bench is 3 feet, and each bench should be provided with cupboards underneath. In these cupboards shelves should be fitted, but one or two ought to be left entirely without shelves, so that tall pieces of apparatus may be stowed away conveniently The benches in the laboratory should be allocated systematically to their respective uses, and they ought not to be used indiscriminately for any operation which may be in process The following hints as to the fitting up and use of the working benches may be useful —

5. Water Bench.—A fairly large bench is convenient for all operations involving the use of gas and water A main gas pipe should be carried the whole length of the bench, provided with taps at convenient distances apart A water main, with similar branch taps for the supply of condensers, etc , should also run the whole length of the bench The water bench might be covered with sheet lead, as it is far more convenient to clean up a lead-covered bench than one not so covered, and, again, when acids are spilled, and accidents generally happen, a lead-covered bench suffers less than one destitute of this protection The water bench should be reserved for operations involving the liberal use of gas and water—these operations are many, and it is convenient, for instance, to have a properly fitted-up water bench when engaged in the testing of feed water, water analysis generally, and the testing of works effluents

6. Gas Bench.—On this bench should be arranged the pieces of apparatus used for heating, igniting, and calcining operations A liberal gas supply should be ensured, and as large a pipe as possible should be carried along the whole length of the bench, with branches at frequent intervals for connecting to burners, furnace, water bath, sand bath, water ovens, etc.

7. The Burette Table.—This should be placed in front of a window, so that in volumetric analysis the end-points of the colour reactions may be clearly observed. Some of these end-points in colour tests may be easily mistaken unless a good light is available, and especially is this the case in such work as Nesslerising in the estimation of ammonia, and also in estimating minute quantities of iron in water Volumetric analysis cannot be satisfactorily accomplished in a bad light, and for this reason the burette table should certainly be placed in the best lighted part of the room.

8. The Balance Table.—In the standard works on chemical analysis a rule is laid down that the balance should be placed in a separate room, and that on no account should it be kept in the laboratory This is all very well in theory, but in practice it is in many cases an almost unattainable state of things. There are few works laboratories where a separate room is available for use as a balance room. In many instances the chemist has to be satisfied with a very small room indeed for the purposes of his laboratory work proper, and if he were so ill-advised as to demand the use of a separate apartment for weighing operations, there is very small chance indeed that his demands would be granted Further, it is often the reverse of convenient that the operator should have to run from one room to another for the purpose of conducting his weighing It would consume too much of his precious time, and this, with the considerations of space and other minor details, render it almost impossible for him to exercise that amount of care for his most delicate instrument, which he fully recognises to be advisable Failing the separate balance room, the balance table should be placed, therefore, in a corner of the laboratory which is as far removed as possible from evaporating apparatus Acid fumes, as is well known, are eminently injurious to delicate instruments such as the balance, and all possible precautions should be taken to protect the instrument from such deleterious influences It is further advisable that the table be not too high; a height which is convenient for the operator to work at *when standing* is, in many cases, more convenient than a table placed for weighing with the operator *seated.*

9. Sinks.—Sinks are a necessity in a properly-equipped laboratory This is by no means a superfluous remark, and it is essential that at least one should be provided This should be placed in a convenient position—in the centre of one of the working benches, if possible A lead-lined well is the best form, for this has several advantages, one of the greatest being that if a piece of apparatus be set down roughly there is less fear of a breakage. Another point to observe is that the pipes leading to the drain should be easily accessible In case of an obstruction this is a facility which will be much appreciated.

THE SELECTION OF APPARATUS.

10 Weighing Apparatus—The most important piece of apparatus in any laboratory is, of course, the balance. Weighing operations are the basis of very many commercial as well as more abstruse analytical processes. It is frequently the case that upon the resultant weight of a precipitate depends the accuracy of a test which may decide the value of goods worth a very large amount, and, this being so, it is in the highest degree necessary that, for purposes of commercial testing, an accurate balance should be available

While dealing with weighing operations and the instruments used in such work, we may consider the need not only of a chemical balance, as it is usually termed, but of weighing apparatus generally It is advisable, then, that several balances of greater and lesser degrees of delicacy be provided. For the general purposes of laboratory work, and for commercial testing generally, balances of several degrees of sensitiveness are useful, but as the requirements of different conditions vary, and as in some cases an approximate weighing is all that is required for the purpose of a commercial test, we shall consider not only the apparatus known as the "chemical balance," but also other balances which may be in demand for various purposes

As has been previously indicated, many of the operations described here might not, strictly speaking, come under the precise head of exact analysis In commercial testing it is often the case that precise analysis is not demanded, and for the purposes of many tests it is not requisite—for example, that a weighing operation should be conducted as far as a tenth of a milligramme In the testing of wood pulp for moisture, where a fairly large sample is taken, as is generally the case, it is not necessary to weigh finer than a tenth of a gramme Again, in a commercial laboratory, there are many experiments which would be carried out with profit, if it were convenient to weigh, with a fair degree of accuracy, a moderately large quantity of raw material and the resultant finished products. Experiments with a few grammes of a given substance are very often not so useful as experiments with a few pounds, and, therefore, while we are considering the requirements of a commercial laboratory, we may just as well take into consideration the possible requirements for the purposes, not only of commercial testing, but of commercial experiments, although it is not proposed to deal with this latter subject in the present volume It is, no doubt, improbable that all the readers of this book will lay themselves out to follow the whole of the suggestions which are made or to procure the whole of the apparatus indicated, but in case at any time information is required as to the best appliance to procure for any specific test, whether analytical or experimental, they will, generally speaking, find within these pages the particulars required as to the best instrument or piece of apparatus, the name of the manufacturer from whom a reliable article may be obtained, and the probable cost at which it can be procured. All this is useful information, and these lines will be followed when indicating the necessary apparatus for each test.

11. The Rough Balance.—This is a comparative term. In the average works—not the works which is already possessed of a chemist and a laboratory, but the works which is as yet innocent of either—it is not infrequently the case that when a delicate weighing is required no suitable "pair of scales" is forthcoming. Suppose a few ounces of material have to be accurately weighed for the purpose of a test, it is often found that the most sensitive appliance in the place is the letter balance, which will not weigh to less than a quarter of an ounce. Now, assume that it is required to estimate the percentage of moisture in any substance; a quantity, say a few ounces, is weighed off, dried, and weighed again, and the percentage is calculated upon the figures so obtained. In order to judge of the probable accuracy of this test, we must remember that the weighings have in each case been conducted to within a quarter of an ounce. In an ounce there are 28·35 grammes, not to go further than the second point of decimals, so that a quarter of an ounce contains 7·09 grammes. Now suppose that with this rough weighing the percentage of moisture was found to be 24·9; assume further that a second test is made, weighing in this case with gramme weights, and to a tenth of a gramme. In the first case the moisture would be returned as 24·9 per cent, and in the latter case the test would actually indicate *over 30 per cent.* This is a mild example of the errors which are introduced into testing when only rough apparatus is available. Many articles of commerce are sold with the understanding that they are not to contain more than a certain percentage of moisture, and when thousands of tons per annum are consumed, as in the case of wood pulp—to which we shall specially refer later on—it is not difficult to see that the consequences of inaccurate testing may be very serious.

For commercial purposes two balances are necessary—a rough balance and an analytical balance The first should be a strong and simply-arranged one capable of carrying about 3,000 grammes, and which will turn with this load to 05 gramme. A balance which answers these requirements is supplied by Messrs F Jackson and Company, of Cross Street, Manchester, and is admirably adapted for many of the operations of commercial testing It is strongly made, the beam is of aluminium, the pans are removable, and it is very sensitive in use Such a balance is useful in a great many commercial tests, and, further, as has already been pointed out, it can be employed for a whole host of other purposes in the commercial laboratory The price is £2 12s 6d

12. The Analytical Balance.—It is a common opinion that any kind of apparatus is good enough for "works tests." There is no greater fallacy A works test ought to be thoroughly reliable, and it is impossible to make accurate tests unless accurate apparatus is provided. For weighing operations the rough balance, as it has been here termed, is sufficiently accurate, but for many tests involving exact weighing it is essential that a much more delicate instrument should be available

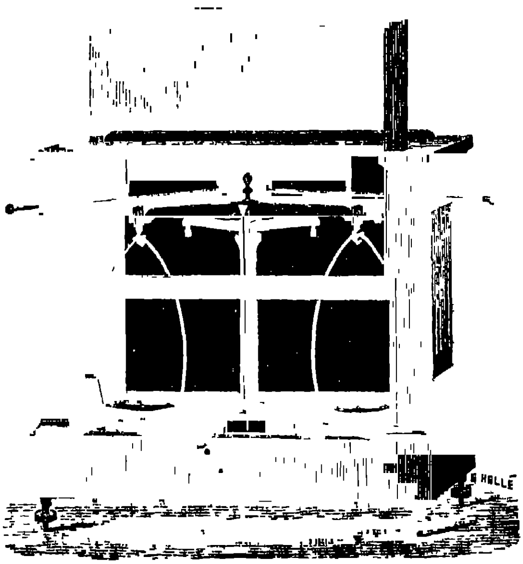


FIG. 1 —THE ANALYTICAL BALANCE.

A chemical balance is a very beautiful and an extremely sensitive instrument, and it is therefore expensive Balances may be obtained of many degrees of sensitiveness, and of many degrees of cost, varying from £5 to £25 It is not necessary for the purposes of commercial testing that a very expensive instrument be purchased A balance which will turn to a fifth of a milligramme when carrying a load of 100 grammes in each pan is illustrated in Fig 1. This balance is well adapted for use in all the tests described in these pages. It is supplied by Messrs. Townson and Mercer, of 34, Camomile Street, London, E.C., at the price of £8 10s complete Cheaper balances may be obtained from the same firm, whose catalogue, or that of Messrs Baird and Tatlock, of 14, Cross Street, Hatton Garden, London, E C , should be consulted

So far as cost is concerned it is extremely moderate, and no manufacturer or firm desiring to economise, or to exercise that control over their buying and manufacturing operations, which is herein advocated, should demur to equipping his laboratory with such a balance. The cost may seem high to the non-scientific mind, even of what is, after all, a very modest balance, but it may be taken for granted that such a piece of apparatus will in a very short time, if used for the purposes of commercial testing, not only pay for itself, but effect a great economy in many directions in any works or establishment where raw materials, such as chemicals, fuels, drysalteries, and the like, are bought and consumed in large quantities The manipulation of the balance will be dealt with later on in this chapter.

13. Weights.—The commercial laboratory should be well equipped with reliable *gramme* weights. For the analytical balance these are, of course, indispensable, and a thoroughly good set should be purchased with the balance. A convenient range is from 100 grammes down to 1 milligramme, with riders. Such a set—of Oertling's make—in box, with forceps complete, will cost from £2 to £2 5s For use with the rough balance, a set of gramme weights, ranging from 1,000 grammes to 1 gramme, in wood block, may be obtained for about 10s. For the smaller divisions a set of cheaper weights, ranging from 50 grammes to a milligramme, may be obtained for about the same amount These two sets are really necessary, and should certainly be procured. Cheap weights can be obtained at a very low cost, but accuracy is sacrificed; and in analytical work, even in commercial laboratory, the economy of a few shillings must not be considered when selecting weights, whatever may be permitted in respect to other apparatus.

14. The Use of the Balance.—There is no operation more important in chemical analysis than that of weighing, and there is no instrument which needs greater care than the chemical balance. It might also be added that there is no instrument which is so carelessly used by inexperienced operators. A balance is very quickly spoiled by careless treatment, and at the very outset it should be understood that extreme care must be exercised in conducting weighing operations. No doubt many of the readers of this volume will have had some amount of practice in weighing with the balance; but, as these instructions are intended more particularly for the use of the inexperienced in such matters, it will be advisable to go into the question somewhat in detail.

Examine the balance carefully, and see that the knife-edges at each end of the beam are in position. See that the instrument is levelled properly by means of a spirit level, with which the balance should be fitted, and, if necessary, adjust the levelling by means of the milled screws at each corner of the case. Now turn the knob in the centre of the balance, and very gently release the supports, causing the beam to oscillate. This movement must be accomplished gently, and, if the beam does not move, return the knob to its former position and repeat the operation. If necessary the door of the case may be opened and closed quickly, the movement of air will then be sufficient to start the oscillation of the beam. Now watch the vibration of the pointer along the scale; at each oscillation the movement will become less, and when four or five divisions only are reached on each side, count them; the first reading should not be recorded. Suppose that the pointer moves five divisions on the left of the scale and three on the right—it is evident that the two scale pans are not in equilibrium. Open the balance case, and, by means of a soft brush, remove any dust which may adhere to the pans and stirrups of the balance. Having brushed the scale pans free from dust, once more close the case, and again release the pan supports. Watch the oscillations closely, not taking the first reading, but allowing the pointer to vibrate several times on each side of the scale before counting When the beam takes a long initial swing, it is a good plan to wait until the oscillations reach four or five divisions only

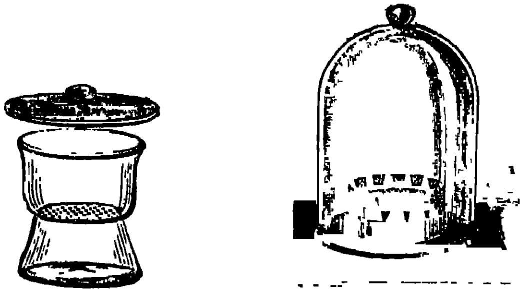
There are one or two precautions which must be observed in using the balance in addition to those already mentioned. In starting the beam in vibration, a very cautious rotation of the milled screw is essential. Turn the screw the least possible touch, and *very* gradually release it, *feeling* the condition of the beam as the screw is turned Again, in arresting the vibration of the beam, it must be allowed to approach zero before checking by means of the screw. If the beam has been swinging violently, it must be allowed to come almost to rest before the screw is turned; then, when the pointer is at zero, a very gentle revolution will completely arrest the motion A shallow vessel containing solid chloride of calcium, or strong sulphuric acid, should be kept in the balance case, in order to keep the atmosphere of the balance case in a dry condition This should be frequently renewed

If it is found, after brushing the pans, beam, and stirrups of the balance, that the beam is still out of equilibrium, an examination should be made Dirt may be caked on the pan, in which case it must be very carefully removed, and recourse should not be made to any of the adjusting screws until the operator is quite satisfied that the beam and pans are quite free from dust and dirt When, however, it is found to be impossible to secure equilibrium otherwise, attention should be given to the adjusting screws At each end of the beam will be found a small milled-headed screw working along the beam Assuming that the pointer still traverses more divisions on the left than on the right, it is evident that the right-hand pan is too heavy, and, accordingly, a fraction of a turn may be given to the screw on the right-hand end of the beam, turning it towards the centre of the beam so as to lighten the load. Now once more release the beam, and continue this adjustment until the divisions on the scale are equally covered by the pointer. The adjusting screws should never be used until it has been proved that the levelling of the balance is correct, and that the beam, pans, and stirrups are quite free from dust.

Assuming that the balance is now accurately counterpoised, an actual weighing operation may be performed, and the following instructions may be followed in all cases when the balance is brought into requisition :—

15. Directions for Weighing.—The first rule to be observed in weighing is that no body must be placed upon the scale pan in a heated condition. It is essential that the substance to be weighed shall be cooled (if it has been previously heated) to the temperature of the laboratory before it is weighed off. In many operations it is necessary to weigh bodies which have previously been dried or ignited. When this is the case the desiccator must be used Suppose, for example, that a platinum dish has been cleaned, dried in the water bath, and is now to be weighed—the dish must first be cooled in the desiccator.

16. The Desiccator.—Two forms of this piece of apparatus are illustrated herewith. They are both supplied by Messrs Townson and Mercer, and are made in different sizes. A good size for Fig. 2 is 6 inches diameter, which costs 3s , Fig 3, 6½ inches diameter, costs 6s The first is very useful for cooling small articles such as crucibles, etc , while the latter is more convenient for use where larger vessels such as wide clock glasses are being used A convenient plan is to provide one of each pattern, in which case it is possible to cool one vessel while another is being weighed; then, by the time the first weighing is finished, the second object is ready for the balance. The lower part of the desiccator shown in Fig 2, and the dish of Fig. 3, must be kept supplied with calcium chloride or strong vitriol, which should be frequently renewed.



FIGS 2 AND 3 —DESICCATORS.

Assuming that the body to be weighed has been cooled in the desiccator if necessary, it may now be transferred to the balance pan. The object to be weighed should always be placed on the left-hand pan of the balance, and the weights on the right-hand pan. This order should be invariably adhered to.

Open the box of weights, and, using the forceps which will be found therein, take out the 100 gramme weight and carefully transfer it to the right-hand pan; then, without closing the door of the balance case, slightly turn the central screw. It will, no doubt, be found that 100 grammes is much too heavy, and if the beam were released suddenly with this weight upon the pan it would be jerked up violently, and, perhaps, be thrown off the knife edges; but if the mere fraction of a turn be given, and the pointer closely watched, it will be instantly seen that this weight is too great. Therefore take off the 100 gramme weight, replace it in the box, and substitute the 50 gramme, once more cautiously turning the screw very slightly. If this also is too much, substitute the 20 gramme weight, which may be too little. Now add the 10 gramme weight, and follow on with the smaller ones, until the dish seems to be nearly balanced. We will now assume that we have on the weight pan the following weights: 20 gramme, 10 gramme, 2 gramme, 1 gramme, 0·5 gramme, 0·1 gramme, 0·02 gramme, and still the dish is heavy. We must now use the rider. This will be found in a separate division of the weight box, and it must be placed upon the hook of the rider slide at the top of the balance case. Many balances are fitted with two riders, but one only is necessary for general work—for the work described in this volume, at all events. The rider, when placed at the extreme right-hand end of the beam, is equal to 1 centigramme—that is, 0·01 gramme. Now try the beam once more with the rider at the end; it is too much. Slide it along to the middle division—each full division is equal to 0·001—that is, a milligramme; so, assuming that the beam is counter-poised with the rider in the centre, then the worth of the rider is 0·005, and the weight of the dish is 33·625 gm.

The final trial of the beam before reading off the weights must be very carefully made. The pointer should be allowed to swing several times, and it must oscillate an equal number of divisions on each side before the weight is accepted as correct. Then when the beam has almost stopped swinging, and the pointer is at zero, gently arrest the beam, and proceed to count up the weights.

In reading the weights the following is a safe course to take: The operator should practise reading off the *spaces* in the box, so that it will be quite an easy matter to reckon the total weight on the balance pan, not from the weights themselves, but from the vacant spaces in the box. Having done this read the weights on the pan, commencing with the largest, and jotting down the value of each one as it is examined. After a time facility will be acquired in reading from the spaces. A third check may now easily be made, and that is to read the weights a third time as they are lifted from the pan to the box. This course of procedure in weighing will obviate many blunders, and, as the operation of weighing is perhaps the most important one in chemical analysis, it is well to indicate here the most correct and safest manner of conducting the operation.

17. Specific Gravity Apparatus.—The specific gravity of liquids may be ascertained (1) by means of the hydrometer, (2) by the use of the Westphal specific gravity balance, and (3) by actual weighing on the chemical balance.

The Twaddell Hydrometer.—For the purposes of commercial testing the Twaddell hydrometer is very frequently employed, and is here illustrated. The instrument is used as follows: We will assume that it is required to ascertain the specific gravity of a solution of sulphuric acid. Take a cylinder, which must be higher than the hydrometer—a test glass can be obtained with the apparatus. Fill this to within an inch of the top, and immerse the hydrometer in the liquid. Twaddell's hydrometers are supplied in sets of six, ranging from 0 to 170° Twaddell. Take No. 1, and immerse it in the liquid to be tested; it at once bobs up to the top, the liquid being far too heavy. Repeat the operation with No. 2; it is still too heavy. Repeat the trial with No. 3 and No. 4. With No. 5 the spindle rises very slowly, and at last comes to a standstill at 136°. The liquid then registers 136° on Twaddell's hydrometer. In taking a "Twaddell," as it is termed, care must be observed that the instrument is kept away from the side of the trial glass. The hydrometer will often tend to stick to the side of the glass, and a reading must not be made until the spindle is [illegible] the centre of the vessel. [illegible]

Messrs Baird and Tatlock, of 14, Cross Street, Hatton Garden, London, E.C. This is a special pattern, which will not roll over or revolve in the liquid to be tested. The price is 1s. 6d each

To convert the degrees on Twaddell's scale to specific gravity multiply by ·005 and add 1·000 For instance, 24° Tw = 1·12 s g

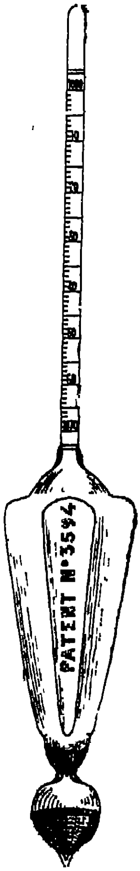


FIG 4 — TWADDELL'S HYDROMETER.

18. The Westphal Specific Gravity Balance.—The Westphal balance is a very convenient and accurate instrument for ascertaining the specific gravity of any liquid. It is of special service when the volume available for testing is small and it has the additional advantage that the actual specific gravity figure is read direct

The balance is kept packed away in a box when not in use, and it must be carefully set up, as shown in illustration Four sizes of riders are supplied with the instrument, and the method of weighing is as follows —

Before taking a reading see that the balance is properly levelled by means of the levelling screws provided at the base of the balance The point on the end of the beam and that opposite to the beam must be perfectly level, so that the beam is in equilibrium This adjustment must be made, of course, before the jar is filled with the liquid Now fill the jar as shown in the illustration Once more assuming that this is vitriol, as before, the plummet will rise as far as it possibly can, and the index pointer will drop down below the spike on the left Now take the riders, of which there are four The largest rider (of which two are supplied) when placed upon the hook at the extreme end of the beam—above the plummet—is equal to 1·0000; placed in the first notch along the beam to the left, it is equal to 0·9000; placed on the next division, it is 0 8000, and so on. The next smaller rider, when placed on the end hook, is equal to 0·1000, and its value diminishes in precisely the same

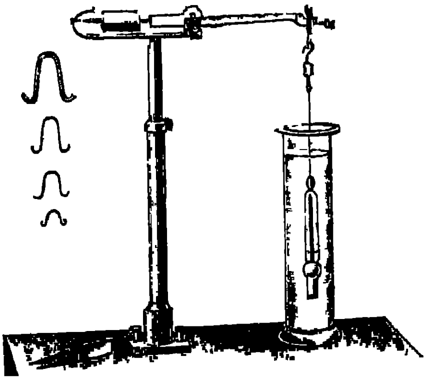


Fig 5—The Westphal Balance.

manner as the larger one The third and fourth riders have values diminishing in the same manner, so that, with the set of four, it is possible to weigh to the fourth place of decimals

With a solution of a similar gravity to the one already tested with the Twaddell, the first of the large riders would be over the plummet, the second on the sixth division of the beam, and, in addition, the rider next in size would be required at the eighth division on the beam The beam will now be in equilibrium, and the specific gravity of the liquid is 1·680

A little practice with the Westphal balance will enable the operator to determine the specific gravity of liquids with speed and accuracy The Westphal is a very convenient, as well as a very reliable instrument, and where it is necessary to determine specific gravities accurately, frequently, and in limited quantities of liquid, it is by far the most convenient method Care must be exercised in setting up the balance, and also in packing it away in its box The plummet must be well washed and dried after each operation, and then, together with its suspension wire, wrapped up in soft paper. The trial jar should also be well cleaned and allowed to dry before being packed away The plummet may be placed inside the jar, and the whole fitted into its place in the box. The price complete, as supplied by Messrs. Townson and Mercer, is £1 7s. 6d

19. The Specific Gravity Bottle.—This is, analytically speaking, the most accurate method of estimating specific gravity; it is also the method which consumes most time in carrying out

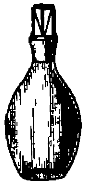


Fig 6.—Specific Gravity Bottle.

There are some instances, however, where it is necessary, and the mode of procedure is as follows·—

The specific gravity bottle is a small stoppered flask containing 100 grammes of water at 60° F., when filled quite full. Clean the bottle, dry in the water oven, cool, and weigh carefully on the analytical balance. Now fill the bottle with distilled water—fill it almost to the top. Take the stopper and examine it; it will be noticed that it is bored with a very fine hole right through Insert the stopper in the neck of the bottle; the water will, of course, come through the hole and run over the neck. With a little practice, however, it will be possible to fill the bottle to such a point that only a little liquid will find its way through the stopper, and this must be absorbed with a piece of filter paper Carefully remove all moisture from the outside of the bottle and weigh again, noting the weight. Now empty the bottle, and rinse it out with a little of the acid solution to be tested, fill in precisely the same manner as with the distilled water, and, after again removing all traces of moisture from the outside, weigh once more The specific gravity, as obtained by this process, is ascertained by the following calculation —

$$\frac{\text{Weight of 100 c. c. acid}}{\text{Weight of 100 c. c. distilled water}} = \text{sp. gr}$$

In using the specific gravity bottle it need scarcely be pointed out that great care must be exercised when working with acid and other corrosive liquids. Unless all traces of such liquids be removed from the outer surface of the bottle before placing it on the balance pan, the pan will certainly suffer, and, for this reason, unless the gravity is required with great accuracy, the Twaddell and the Westphal are most frequently used. For commercial purposes the balance is very rarely used At the same time it is useful to be able to take a gravity by actual weighing

20. Water Ovens.—For all laboratory purposes, where the temperature required is not over 212° F. (100° C), a water oven should be used Water ovens are all constructed on the same principle—they consist of an inner drying chamber surrounded by an outer jacket. In the space between these water is placed, and heat applied by means of a gas burner . By this means the heat of the drying chamber cannot, of course, exceed that of boiling water—that is, 212° F., and as, for many laboratory operations, this temperature is sufficient, the water oven is by far the most useful piece of drying apparatus.

In Fig. 7 is shown a special form of oven, made by Messrs. Townson and Mercer, which consists of the oven proper, combined with a cistern for maintaining an automatic supply of water without lowering the temperature of the drying chamber. This is an exceptionally useful arrangement, and obviates the necessity of maintaining a constant watch over the water level

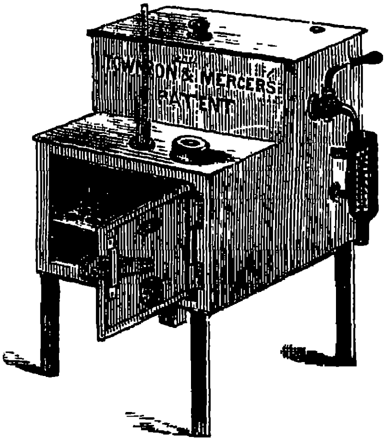


Fig. 7.—Water Oven

It is not an uncommon occurrence for a water oven to boil dry, and when the attention of the operator is drawn to the apparatus he sometimes finds that the solder has been melted out of the joints, and that the oven is ready for the repairers. All possi-

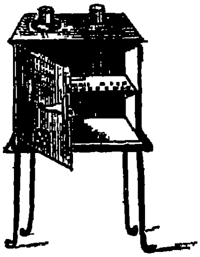


Fig. 8.—Water Oven.

bility of such an occurrence is removed in the oven under notice, and, moreover, as the feed water is heated before its entry into the water jacket, the temperature of the drying chamber is kept constant. This, of course, is not the case with the [illegible] arrangement, in which cold water is often poured into the jacket. A convenient size for such an oven is 6⅞ in. by 7¾ in. by 6¼ in., and the price is 40s. A larger size, 9 in by 9 in by 9 in, is also supplied at 60s.

The ordinary form of water oven, illustrated in Fig. 8, consists of a copper drying-chamber surrounded by a water jacket, as in the last described. In this pattern of oven the water supply is not automatic, but for many purposes a small oven, in addition to a large one, is often a convenience, besides which, the low price of an oven of this type places it in the reach of any laboratory Messrs. Townson and Mercer supply this oven, size 6⅛ in by 6¼ in. by 5¼ in Price 21s.; with stand, as shown, 2s. 3d. extra.

21. Hot-air Ovens.—For some purposes it is necessary to obtain a temperature of over 100° C. (212° F.) With a water oven this is not possible As a matter of fact, few water ovens give a temperature of more than 96° C, and when it is absolutely necessary that 100° C is to be maintained, it is necessary to use a hot-air oven The difference between the air oven and the water oven is that in the water oven the water jacket is the medium through which the heat is conveyed to the drying chamber; in the case of the air oven the water is dispensed with and hot air passed through the jacket, thus enabling any desired

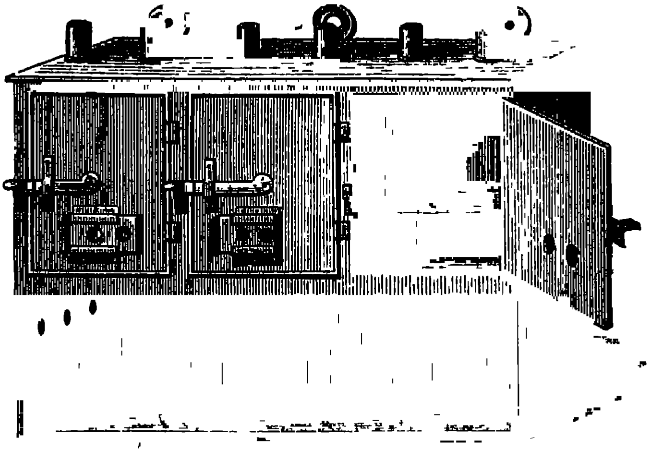


FIG 9.—SPECIAL AIR OVEN

temperature to be obtained in the drying chamber. Three hot-air baths are here illustrated; they are all supplied by Messrs. Baird and Tatlock The largest is a very useful piece of apparatus, and is convenient for many operations. As shown, with three compartments, each 9 in by 7 in by 6¼ in., with a lower chamber of sheet iron, the price is 50s. Fig. 10 is a useful little hot-air oven; 5 in by 5 in is supplied at 18s 6d.

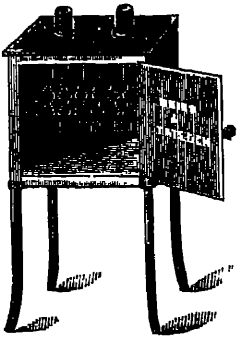


FIG 10.—ORDINARY AIR OVEN

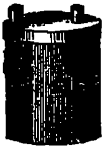


FIG 11.—SMALL CIRCULAR AIR OVEN

The small circular bath, shown in Fig. 11 (price 7s. 6d), is useful for many purposes where it is necessary to use very high temperatures For drying wood pulp, special water and air baths are used, but these are constructed on the same principles as the ordinary laboratory ovens, and differ only in size

22. The Water Bath.—For many purposes in testing, such as the evaporation of liquids and the dissolving of substances in liquids, the water bath is required. It is made in several forms, of which that shown in Fig 12 is the most convenient.

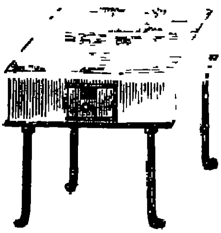


FIG 12.—WATER BATH

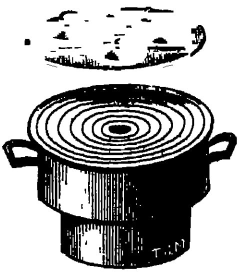


FIG 13.—CIRCULAR WATER BATH.

It is made of copper, with holes for basins, beakers, etc , together with a drying drawer For drying precipitates, and for evaporating purposes generally, and especially in water analysis, this form of bath is a very useful piece of apparatus In copper, 12 in. by 9½ in by 4 in deep, this form of water bath costs about 30s A cheaper form of bath is shown in Fig. 13, which, with movable rings, is also supplied by Messrs. Townson and Mercer. Price 15s in copper, 8 in. diameter

23. Gas Burners.—For laboratory purposes the different forms of gas burners supplied by Messrs. Fletcher, Russell, and Company, Limited., of Warrington, cannot be surpassed. As it is convenient to have particulars of the most useful forms of these gas burners, several are here illustrated.

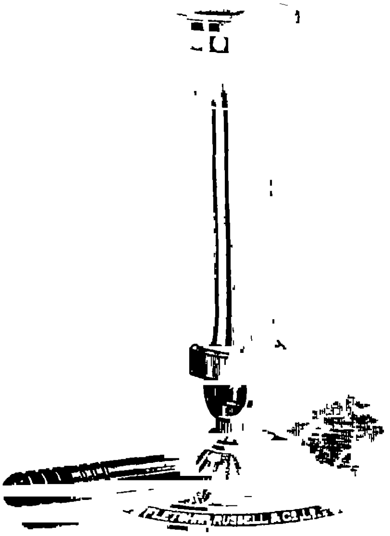


FIG 14 —IMPROVED PATENT BUNSEN BURNER.

FIG 15 —LABORATORY BUNSEN BURNER.

24. Bunsen Burners.—The Laboratory Bunsen (Fig 15) is a very useful burner. As shown, with rose, to burn 4 ft. of gas per hour, the price is 2s. 6d.; without rose, 9d. less The patent safety Bunsen (Fig. 14) is the best form for boiling purposes. This burner will not light back, and the flame can be turned down to the merest flicker. The safety burner is made in a variety of sizes, and at prices from 3s 6d to 5s 9d. each, including brass stand

25. The Sand Bath.—This bath, being easily regulated, is of great use in laboratory work generally. There are many processes in analysis where a constant and efficient source of heat is required which cannot be obtained by means of the water bath For evaporating liquids to dryness, and for many other purposes, the sand bath is a great convenience

The stand for vessels will carry flasks, porcelain dishes, or flat-bottomed vessels of any size with perfect steadiness. The vessels can, by lifting the cylinder, be placed at different heights above the flame, which will burn steadily when turned down to its lowest point. When in use, the shallow tray, which fits on top of the cylinder, is filled with sand, and the flask, basin, or beaker containing the liquid to be evaporated is placed in or on the sand, a steady evaporation, without bumping, being the result The bath is supplied by Messrs. Fletcher, Russell, and Co. in various sizes, at prices from 4s to 17s. each.

26 The Muffle Furnace.—There are few works' labora-

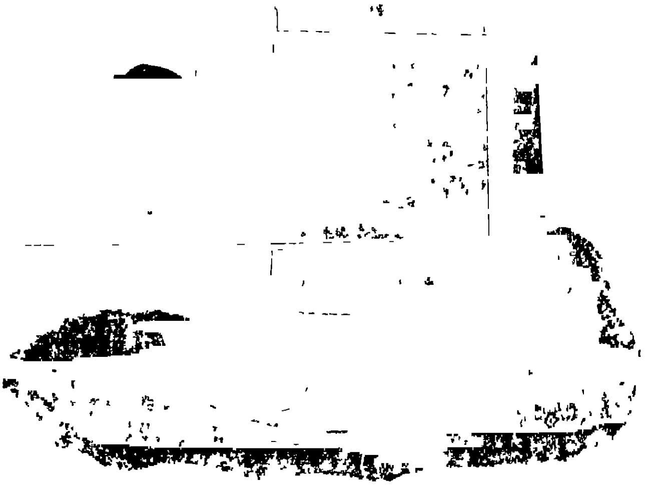


Fig 10 —Muffle Furnace.

tories which are equipped with a muffle furnace. For use in the testing of fuels, the analysis of various raw materials which require the determination of ash, the ignition of precipitates and residues of all descriptions, the muffle furnace is a great convenience. If possible one of these furnaces, as illustrated, should be installed Fig. 16 gives an external view, and Fig. 17 shows the internal arrangement. In working this furnace, a good supply of gas is necessary If the gas supply is deficient, the furnace can be worked at a lower heat by partially closing the top of the chimney, or by working without chimney The points of blue flame are always visible when the burner is looked into sideways, unless the gas supply is too small to work the furnace satisfactorily. To light the burner, without removing the top part of the furnace, put a lighted taper through the burner casing up between the grooves in the burner plate, and then turn the gas on slowly If the furnace is hot, it may be necessary to cover the air opening round the gas entrance of the burner, to prevent the flame descending through the gauze at the moment of lighting. The burner should be kept clean. The price of furnace 2 in. wide, 2 in. high, and 4 in. long inside muffle is 40s The whole of these burners are supplied by Messrs Townson and Mercer.

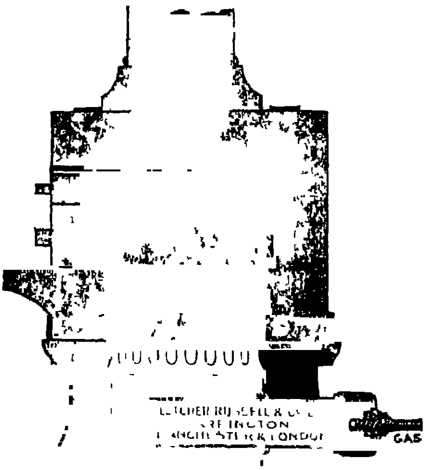


Fig 17.—Muffle Furnace (Showing Internal Arrangements)

1283
541.26
N01

27. Apparatus for Volumetric Analysis.—In volumetric analysis the principal pieces of apparatus used are the measuring flask, the burette, and the pipette The apparatus is illustrated as supplied by Messrs Baird and Tatlock

28. The Measuring Flask.—Measuring flasks, with ground stoppers, should be obtained in the following capacities: Litre (1,000 c c), half-litre (500 c.c.), 250 c c, 100 c c., and 50 c.c, costing, respectively 2s 3d, 1s 9d., 1s 6d, 10d, and 8d each.

Plain flasks may be obtained, but for these rubber stoppers are required, and stoppered flasks are the most serviceable. In using measuring flasks, it must be remembered that the capacity is reckoned at 60° F., and all operations must be conducted on this basis. For instance, if it is desired to filter a liquid into a flask, make the filtrate up to 500 c.c., and then titrate a portion of this quantity; the filtration may be done while hot, warm water used for washing, and then the volume made up with cold water The contents of the flask may now be quite warm, and, before the final adjustment is made, the flask and its contents must be cooled This cooling may, of course, be accelerated by allowing cold water to flow over the flask. In making the final adjustment to the mark, the lower portion of the meniscus—the curve made by the level of the water in the neck must just touch the mark; the top of the curve must not be used.

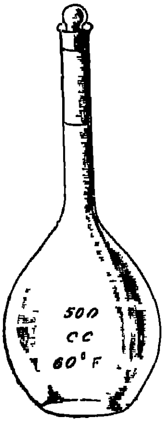


FIG 18.—GRADUATED FLASK

29. The Pipette.—This is an instrument for measuring off a definite quantity of a liquid when filled to a mark on the upper end of the tube. The most useful sizes of pipettes are, 50 c.c., 25 c.c., and 10 c.c. Pipettes are of two kinds—the bulb pipette for delivering a given volume of liquid, and the scale pipette, by means of which any quantity, from one-tenth of a c.c. to 10 c.c., may be delivered. The bulb pipette is used as follows. Fill the pipette by sucking up liquid until the mark on the stem is reached. In practice the stem should be filled up to about an *inch above* the mark. The filling is best done by holding the pipette in the right hand, with the first finger closing the suction end. Now remove the finger, apply suction, and when the liquid has reached the desired height quickly remove the lips, and as quickly apply the first finger again, pressing tightly upon the end of the tube. Now hold the pipette up to the light (still keeping the end closed with the finger), and release the pressure *very slightly;* a drop or two of the liquid will flow out. Allow this dropping to go on until the level in the tube—the bottom of the ring, not the top—coincides with the mark on the stem. The pipette now contains 50 c.c., and the contents may be delivered into any vessel in which the subsequent treatment is to be carried on. The price of bulb pipettes is, 10 c.c. 6d., 25 c.c. 9d., and 50 c.c. 11d.

30. Scale Pipettes.—Scale pipettes are very useful for delivering small quantities of liquids. The pipette is filled to the mark indicating 10 c.c., and, assuming that 1 c.c. only is required to be delivered, the level of the liquid is allowed to

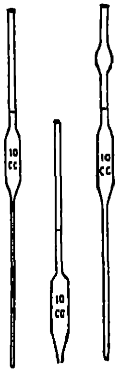


FIG. 19.—BULB PIPETTES.

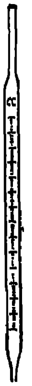


FIG. 20.—SCALE PIPETTE.

sink from 10 to 9, when 1 c.c. has been delivered. A scale pipette, with a capacity of 10 c.c. and graduated in tenths, costs 1s 4d.

31. The Use of the Pipette.—In emptying a pipette, it is possible to introduce an error by improper delivery The instrument should be used as follows: First rinse out the pipette with a little of the liquid which is to be measured When the stream of rinsing liquid has stopped, allow the last few drops to fall, rest the pipette a few seconds on the side of the vessel, and then shake gently. Two or three drops will detach themselves. Wait a few seconds, and again shake, after which consider the delivery completed. Now fill again for the measurement of 50 c.c. Empty the pipette as detailed above, *allowing the same length of time to elapse in the detachment of the last few drops*, and carry out the process in exactly the same manner whenever using a bulb pipette.

32. The Burette.—Three forms of burette are illustrated. Mohr's burette, with jet and pinch cock (Fig 22), which is suitable for liquids such as acid, soda, etc; in fact, for use in acidimetry and alkalimetry The same burette, with stopcock (Fig. 23), should be used for solutions, such as permanganate of potash and other reagents which would attack the rubber of

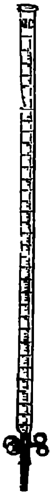


FIG 21.—MOHR'S BURETTE WITH PINCHCOCK.

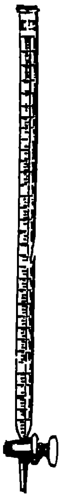


FIG 22. MOHR'S BURETTE, WITH TAP.

the pinchcock burette. The third form, Bink's burette (Fig. 24), is also a very useful instrument, particularly for such operations as the estimation of hardness in water

In using the burette, the following precautions must be observed First rinse out the burette with clean water, invert it in the burette stand, and allow it to drain. Then run into the tube a little of the solution to be used, and, inverting the tube, with the end closed by the palm of the hand, well rinse the burette, finally allowing the liquid to run through the stopcock or pinchcock Now fill up to the mark with the solution, reading, as already observed, from the bottom of the meniscus, and deliver the solution slowly into the beaker The same method of procedure is, of course, followed when using the burette with glass stopcock, but in this case the tap must be smeared lightly with grease before using Cost of Mohr's burettes, with jet and pinchcock, 50 c.c graduated in tenths, 3s. 6d ; the same burette, with glass stopcock, 4s. 3d.

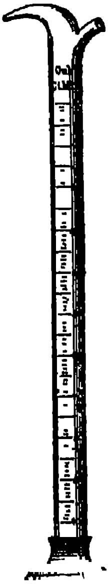


FIG. 28.—BINK'S BURETTE.

33. Bink's Burette.—In using Bink's burette, first smear the mouth of the delivery tube lightly with grease, in order to prevent drops from running down the side of the burette Rinse out with a little of the solution to be used, and again fill up to the mark To deliver the liquid, take the burette out of the foot, and hold it in the closed hand, with the end of the forefinger pressed tightly over the mouth. Now invert the burette so as to introduce the delivery jet into the flask, bottle, or beaker in which the titration is being made, and slightly release the pressure, when the solution may be delivered, drop by drop, at any desired speed Bink's burettes are generally graduated in tenths of a c c , and contain up to 50 c c. Price, with wooden foot, 4s 6d

34. **Rose's Crucible**—This crucible is used when it is desired to ignite a precipitate or other substance out of contact with air. A rubber tube conveys a current of gas through the leading tube, which fits into the lid of the crucible The gas finds its way underneath the lid of the crucible, and ignites when a burner flame is applied. This crucible is supplied by Messrs. Baird and Tatlock Price 4s.

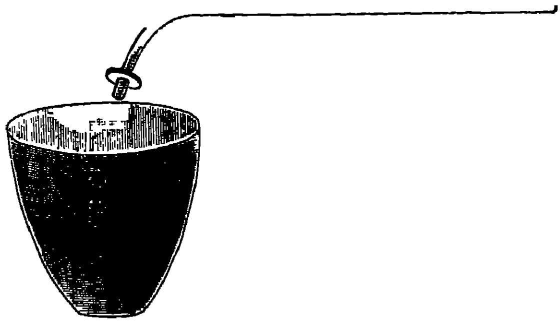


FIG. 24—ROSE'S CRUCIBLE.

35. **Beakers.**—Two kinds of beakers are useful in general laboratory work—the ordinary wide-mouthed beaker and the conical beaker The former is useful for general work, dissolving, boiling, etc., whilst the conical beaker is very convenient for purposes of titration. In some works a flask is used for this purpose, but a conical beaker is preferable. Conical beakers are also useful for many other purposes.

36. **Flasks.**—Bohemian glass flasks of several sizes should be available for general use In the fitting up of apparatus for

various experiments, and for pieces of apparatus such as wash-bottles, etc., they are a necessity.

37. Funnels.—Funnels of three or four sizes, for filtering purposes, and for transferring solutions to flasks, etc., are useful Plain funnels will be found convenient for most operations, though ribbed funnels permit of more rapid filtering when difficult liquids have to be dealt with.

38. Evaporating Basins.—Two or three white porcelain evaporating basins of different sizes are useful for many operations where the volume of a solution needs to be reduced. For tests of chlorine in water, a clean, white basin should be set aside for the purpose, and should not be used for anything else

39. Mortars.—An iron mortar is necessary for the crushing of ores, minerals, and any substance of a refractory nature Two porcelain mortars of different sizes, and an agate mortar for reducing ores to an impalpable powder, are also useful Of course, in the works laboratory, only such apparatus as is absolutely necessary will be procured, but where a little expense is not grudged, money spent on apparatus will be found to be a good investment.

40. Sundry Apparatus.—A good supply of glass tubing of different sizes, glass rod, watch and clock glasses for covering beakers and basins, rubber stoppers and corks, a set of cork borers, a triangular file, and such other small pieces of apparatus should be provided for general use

41. Thermometers.—Both Centigrade and Fahrenheit thermometers should be available—at least one of each kind. A Fahrenheit thermometer graduated to about 300°, and a Centigrade thermometer ranging up to 200°, are useful. Others, graduated in half degrees, and with lesser ranges, are convenient All thermometers should have milk-glass scales; paper scales are unsatisfactory. In taking a temperature, stir the liquid well, with as much of the stem as possible immersed, for a few seconds before reading off.

42. The Wash Bottle.—For many purposes it is convenient to use a fine jet of water, as in washing precipitates, rinsing out beakers, and making up measured quantities of liquid. To fit up a wash bottle, take a good-sized flask with a flat bottom, and fit it with a rubber stopper perforated with two holes. Bend a piece of glass tubing to form an air tube for the lips Into the other hole fit another piece of tubing reaching to the bottom of the flask, and bent over the neck of the flask at the top Take a bit of soft tubing, heat it in a gas flame, and draw it out to form a fine jet; fit this on to the bent end of the long tube by means of a bit of rubber tubing Fill the bottle with water, and, on blowing down the mouthpiece, a fine jet of water may be obtained for any purpose. If hot water is required, heat the flask on the water bath or over a Bunsen burner

43. Filtering—Filter papers of a good make should be used, and several sizes should be kept in stock In fitting a paper, fold it across once, then a second time across itself, keeping the point sharp and well defined Open it out, and fit it into a funnel of such a size that half an inch margin of the funnel is left above the paper. Now wet the paper under the tap, and press it into its place; smooth out any air-bubbles, and proceed to filter Keep the funnel well filled with the liquid Where a precipitate has to be separated, pour off the clear liquid first, then transfer the precipitate by means of a jet of water from a wash bottle

44. Precautions to be observed in using Platinum Vessels—Platinum crucibles or basins must not be used in the ignition of compounds of lead, zinc, antimony, tin, or arsenic For such substances, porcelain crucibles should be employed.

45. Ignition of Precipitates.—Precipitates are ignited in two ways. (1) Apart from the filter paper, and (2) with the filter paper After filtering, allow the precipitate to drain for a few minutes in the funnel, then carefully remove the paper, and spread out on a clean clock glass; tap the glass on the bench to distribute the wet precipitate evenly, and dry on the water bath When fairly dry, remove to the water oven, and continue until quite dry.

(1) *Precipitate and filter paper ignited together*—Fold the precipitate up in the paper in such a way that the precipitate is in the centre. Clean and weigh a porcelain or platinum crucible Into this place the folded-up precipitate, and heat over a Bunsen burner gently. Allow the paper to burn off, then increase the heat, and ignite until all combustion ceases. Keep at a dull red heat for about ten minutes; remove to the desiccator, and weigh when cold Again ignite for a few minutes,

cool, and weigh. If any loss is indicated, ignite again, and obtain a third weighing. Continue the ignition until the weight is constant

(2) *Precipitate ignited apart from filter paper.*—In some cases it is necessary to observe this precaution, where the ignition of the paper might have a reducing effect on a metal contained in the precipitate Dry well in the water oven; prepare a weighed crucible, and carefully transfer the dried precipitate Rub the folds of the paper gently together, letting the precipitate fall into the crucible Brush off the fragments loosely adhering with a soft brush. Now take the filter paper and roll it into a *loose* ball. Wrap around this a length of stout platinum wire, leaving a length of about three inches uncoiled Hold the wire cage over the crucible, placing a sheet of glazed white paper underneath Take a Bunsen burner without the rose, and, holding the flame in a horizontal position, introduce the filter paper. Light the paper, allow it to burn off, and then hold the whole in the flame until completely ignited. Tap the cage on the side of the crucible, and the precipitate with the ash of the paper will fall The ignition is then completed as described above

The Ash of the filter paper —If a good quality of paper is used the ash of each paper will be found marked on each packet. If a very accurate analysis is required, this figure is deducted from the weight of the precipitate + filter paper For approximate tests it is often neglected

STANDARD SOLUTIONS

46.—Standard solutions, upon which the system of volumetric analysis is based, may be either bought, or made up in the laboratory In many works no attempt is made in the direction of preparing standard solutions, and the solutions required are obtained from the laboratory furnishers This is a very convenient method, especially when small quantities of the various solutions only are consumed, but it is often advisable to make up standard solutions in the laboratory, and accordingly simple instructions for the preparations of the principal solutions are here given.

In Sutton's "Volumetric Analysis" we are told that "volumetric analysis, or quantitative chemical analysis by measure, in the case of liquids and solids, depends upon the following conditions for its successful practice: (1). A solution of the reagent or test, the chemical power of which is accurately known, called the 'standard solution' (2). A graduated vessel from which portions of it may be accurately delivered, called the 'burette.' (3). The decomposition produced by the test solution with any given substance must either in itself or by an indicator be such that its termination is unmistakeable to the eye, and thereby the quantity of the substance with which it has combined accurately calculated"

The burettes, pipettes, and flasks used in volumetric analysis have already been described and illustrated The standard solutions most used in the commercial testing are as follows:—

47. Normal Sodium Carbonate.—(53 gm (Na_2CO_3) per litre) Weigh out accurately 53 gm pure anhydrous sodium carbonate, place in a beaker, and dissolve over the water bath in distilled water, stirring constantly with a glass rod until dissolved When completely dissolved, transfer to a litre flask, and make up to the mark, finally adjusting the solution when cold

48. Normal Sulphuric Acid.—(49 gm H_2SO_4 per litre.) Measure off about 30 c c of pure sulphuric acid into a beaker, into which has been placed a little distilled water. Mix well, transfer to the litre flask, and make up to the mark when cold Titrate 10 c.c. of the solution against the sodium carbonate solution already made, and note the quantity required to neutralise the 10 c c. of acid. If more than 10 c c. are required, the acid is too strong, and the solution must be diluted until 10 c c. of acid exactly neutralise 10 c c. of sodium carbonate solution. The acid should be made up a little too strong in the first place, as it is most convenient to dilute the solution 1 c.c. of acid = 0 049 gm. H_2SO_4.

49. Normal Hydrochloric Acid.—(36·5 gm. HCl. per litre) Make a solution of pure hydrochloric acid of 1·020 specific gravity (4° Tw). This will be a solution stronger than normal. Titrate 10 c c. against the standard sodium carbonate solution, and dilute until 10 c.c. of the acid exactly neutralise 10 c.c. of the carbonate solution.

50. **Normal Nitric Acid.**—(63 gm. HNO_3 per litre) Make up a solution of pure nitric acid of such a strength that it exactly corresponds to the standard sodium carbonate solution

51. **Normal Caustic Alkali.**—For this solution pure sodium hydrate made from metallic sodium should be used Dissolve about 42 gm of this in a little less than a litre of distilled water. Titrate 10 c.c of the solution against the normal acid, and adjust until 10 c c of acid exactly neutralise 10 c c. of caustic alkali

52. **Decinormal Potassium Permanganate Solution**—(3·18 gm. per litre.) Weigh off 3·18 gm of pure and dry crystals of potassium permanganate, and dissolve in a litre of water This solution should be standardised by titrating against a decinormal solution of oxalic acid, made by dissolving 6 3 gm. pure oxalic acid in a litre of distilled water.

53. **Arsenious Acid Solution for Available Chlorine.**—(13·95 gm As_2O_3 per litre.) Weigh off accurately 13·95 gm of pure re-sublimed arsenious oxide Dissolve this, in a beaker, with caustic soda solution Add distilled water, acidify with hydrochloric acid when cool, make the solution up to a litre

54. **Normal Oxalic Acid**—(63 gm $C_2O_4H_22H_2O$ per litre.) 63 grammes of pure crystallised oxalic acid dissolved in a litre of distilled water.

55. **Decinormal Nitrate of Silver**—(17 gm $AgNO_3$ per litre.) Weigh off 17 gm of pure and dry crystals of silver nitrate, and dissolve in a litre of distilled water 1 c c of this solution = 0·00355 gm of chlorine.

56. **Standard Solution of Ammonia for Nesslerizing.**—Dissolve 0 0315 gm of pure sublimed sal-ammoniac in a litre of ammonia-free water. 1 c c. of solution = $\frac{1}{100}$ m. gm (0 00001) of NH_3.

57. **Alkaline Permanganate Solution for Water Analysis**—Dissolve 8·0 gm. of crystals of pure potassium permanganate, and 200 gm stick caustic potash in a litre of ammonia-free water

58. **Soap Solution.**—Dissolve 10 gm. pure Castile soap in 500 c.c methylated alcohol and 500 c c distilled water. Allow

the solution to settle for a week or ten days, and check by the chloride of calcium solution.

59. Chloride of Calcium Solution.—Dissolve 1·11 gm of pure dry calcium chloride in distilled water, and make up to a litre

60. Nessler Reagent—Weigh off 35 gm of potassium iodide and 13 gm mercuric chloride Place in a large beaker, and heat almost to boiling. Add a cold, saturated solution of mercuric chloride, adding drop by drop until a red precipitate is just permanent To this solution add 160 gm of stick caustic potash, or 120 gm of stick caustic soda (pure by alcohol), and dilute the whole to a litre Finally adjust by adding a *little* of the cold saturated solution of mercuric chloride, until the solution has a faint brownish-yellow colour—it must not be colourless, 2 c.c. of the re-agent should give a distinct colouration with 1 c c of the standard solution of ammonium chloride (See paragraph 56)

61. Standard Iron Solution—Weigh off exactly 1 gm. of pianoforte wire, dissolve in pure sulphuric acid, and make up to a litre with distilled water Label this "Stock Solution" Of this solution take 10 c c exactly, and make this up to a litre with distilled water. Label this "Standard Solution" 1 c c of standard solution = 0·00001 ($\frac{1}{100}$ of a milligramme) of metallic iron.

62. Litmus Solution.—Powder coarsely 5 gm. of litmus Place the coarse powder in a beaker, add about 200 c.c. of distilled water, and digest for four hours When the sediment has settled to the bottom of the beaker, pour off the clear liquid. To this add a little dilute nitric acid, drop by drop, until the litmus solution is of a violet colour. Pierce a stopper with a hole in the middle, into which insert a piece of glass tubing long enough to reach the bottom of the bottle in which the solution is kept When required, the solution is withdrawn by pressing the finger on the end of the tube, withdrawing the cork and tube, and dropping out as much as is required of the solution. Acids turn litmus solution, or litmus paper, from blue to red. Litmus should not be used where carbonic acid is present.

63. Methyl-Orange.—Dissolve one gramme of methyl-orange in a little methylated spirit Make the solution up to a litre with a mixture of equal parts of methylated spirit and water. Alkalies change the colour of methyl-orange to yellow, in presence of acid the colour is changed to red Methyl-orange is not affected by carbonic acid

64. Phenol-Phthalein.—Dissolve in methylated spirit as in last paragraph When phenol-phthalein is added to an alkaline solution the liquid takes a bright red colour, this colour is removed completely when acid is added Carbonic acid and ammonia affect the use of this indicator

65. Indigo Solution.—Indigo dissolved in strong sulphuric acid and diluted

66. General Reagents.—All the reagents used in testing should be pure Acids and ammonia are generally diluted to one-third strength for ordinary purposes; other reagents are made up into saturated solutions Solids should be kept in tightly-corked bottles with wide mouths All reagents should be plainly labelled

CHAPTER III.

The Testing of Fuels.

67. Fuel Testing.—The commercial testing of fuels must be regarded, not so much as an exact analysis, as a means of comparison of the different sources of supply which are open to the manufacturer. In considering the subject for the purpose of this manual, it is assumed that the need of the consumer is to be able to discriminate, when placing his contracts, between various samples, say, of slack, which may be offered to him. Or, again, he may not be satisfied that he is getting the best value for the money he is continually paying away for fuel. What is his remedy? There is the weekly consumption under the boilers, of course, which ought to show whether his fuel is economical, but he may not have the necessary data even to compare these figures. Again, he may change his supply, and place a contract with another mine, but it does not follow, although that mine may have a reputation for producing better fuel-raising slack than the previous one, that, therefore, his fuel bill will show a decrease A little juggling on the part of the boiler attendants may neutralise any reform which the manufacturer may seek to effect, especially when there is a secret commission paid to the man who has control of the coal burning.

Thus the consumer is driven to the fact that the only way in which to ascertain the real value of his fuel supplies is to resort to chemical analysis of the fuels themselves. Take, as an instance, ten samples of slack, each of which is supposed to be equally good for steam raising It is quite possible that, out of the ten samples, one may show several times more ash than the others This high percentage of ash at once condemns the particular sample. The same considerations must apply with respect to the other constituents—moisture, coke, fixed carbon, and volatile matter

68. Sampling Coal, Coke, &c.—A fair average sample must be carefully drawn This may be best accomplished by taking a shovelful of the slack from various portions of a load, and placing the selected part on one side. A liberal number of such samples should be taken, and a cone-shaped heap formed Now proceed to deal with this large sample by what is known as the process of quartering Flatten the top of the heap, and divide it by a line through, and then another at right angles to the first. The cone-shaped heap of slack is now divided roughly into four quarters Take two of these, from opposite sides, and shovel the selected quarters into a fresh heap, rejecting the remainder altogether Now form a second cone-shaped pile, again flattening at the top. Divide this into four quarters as before (a piece of board is a good instrument for marking the divisions); take two opposite quarters and separate them, again clearing away the rejected portion. Repeat this operation, mixing well each time, as often as may be necessary in order to reduce the original heap of slack to a sample of a few pounds, when it is ready for further treatment in the laboratory. Take the sample obtained as above, and spread it out on a sheet of stout paper on a good large table, and prepare the coal for analysis.

69. Moisture.—The moisture test may be made on the sample before further treatment Well mix the sample, and place a quantity into a large mortar. Crack the lumps, and reduce the whole to pieces about the size of a pea, but do not powder the coal. Now weigh out, on the rough balance, 100 grammes of the roughly-crushed sample. Take a sheet of white glazed paper, and, by folding over the edges and corners, form a simple tray. Place the 100 grammes of coal in this tray, and dry in the water oven at 100° C., weighing in two hours After a time, coal begins to gain weight by oxidation, and the sample should therefore be weighed at short intervals, the lowest weight being the dried weight of the sample. The loss on drying gives the percentage of moisture contained in the coal; for instance, 100 gm. of coal weighs, after drying, 93 24 gm.; the moisture contained in the sample is therefore 6·76 per cent

70. Coke.—After weighing off the 100 gm for the moisture test, further reduce the sample by quartering, after crushing in the mortar, until only a small sample is left—about 200 gm This should be reduced to a very fine powder, and placed in a tightly-stoppered sample bottle, for use in the estimation of the coke, volatile matter, and ash

For the estimation of the coke, weigh off accurately, on the analytical balance, 2 gm. of the powdered sample Dry and weigh a platinum crucible, with lid, into which place the 2 gm. of coal Heat the crucible, with the lid almost, but not quite in its place, over a powerful Bunsen burner, the flame of which should be kept at a constant height In Lunge and Hurter's "Alkali Makers' Pocket-book" a height of not less than 7 inches is specified. The crucible should be supported on a pipe-clay triangle fixed on a tripod stand, the crucible being inclined at an angle. The heating should be continued for a definite length of time A good plan is to heat for four minutes without the lid being quite fixed in position on the crucible, then for a further four minutes with the lid in place In making tests of a number of samples of fuel, the position of the crucible, the height of the flame, and the period of heating must be uniform

After the heating has been carried on for the requisite length of time, remove from the flame, cool under the desiccator, and weigh. Assuming the weighings obtained are as follows: Residual coke, from 2 gm sample, 1·10; thus the percentage of coke will be 1 10 × 50 = 55 0 per cent, while the volatile matter is, of course, obtained by subtraction of the coke percentage from 100—that is, 100 0 − 55·0 = 45 0 per cent.

71. Estimation of Ash—In the estimation of the mineral matter or ash contained in the coal, it is necessary that the sample should be very finely ground If the method of procedure outlined above be followed, the sample, which has been placed in a stoppered bottle, will already have been reduced to a fine powder, and this will be sufficiently fine for the estimation of the ash

Weigh off 1 gm. of the finely-powdered coal. Place this in a carefully weighed platinum dish or crucible, and ignite in the muffle furnace. In the first stage of ignition, the furnace having been heated to dull redness, place the dish containing the coal at the mouth of the muffle, so as first of all to drive off the volatile matter at a low temperature. Now introduce the dish further into the muffle, and keep at dull redness until the ash has assumed a grayish appearance. This may be ascertained by taking out the dish at intervals by means of the crucible tongs, allowing to cool, and stirring about the ash with a stout platinum wire, taking care that any fragments adhering to the wire are brushed off into the dish When the ignition appears to be complete, and the whole of the residual ash has assumed a grayish colour, remove the dish to the desiccator, allow it to cool, and weigh Return the dish to the furnace, resume the ignition for a time, cool, and again weigh. If any loss has taken place, this operation must be repeated until the weight obtained is constant.

In the estimation of mineral matter in coal it is very important that the ignition should be complete. As only 1 gm. of the sample is taken for the purpose of the test, it will at once be seen that, should the ignition not be complete, the error so introduced may quite easily be a grave one. In fact, cases have occurred where, in a works in which this test had very rarely been performed, an incomplete ignition of the sample of coal gave results which were utterly absurd. The works chemist had been instructed to test the slack which was being consumed for steam-raising purposes, and his method of procedure was as follows He took a piece of the coal weighing about 50 gm, and, without any preliminary crushing of any description, he placed the sample into a good-sized crucible, and ignited over a feeble Bunsen burner until he obtained a very uncertain form of residue. When the mineral matter was calculated on the basis of this test, the percentage was so very high that it was plain that the test was utterly valueless, and when it was repeated by a proper method, the difference was so great as to emphasise the necessity for a reliable and accurate test being placed in the hands of works chemists who are only called upon occasionally to test the fuel in the mill; therefore, it is not too much to again emphasise the necessity for ensuring thorough ignition of the sample when estimating the mineral matter in any given coal. A muffle furnace is best suited for the estimation of the ash, but a strong Bunsen burner will answer the purpose; the ignition will, of course, occupy a longer time.

The calculation of the percentage of ash from the figures obtained as above is very simple If 1 gm. of fuel is used for the test, and the residue weighs 0·032 gm, then the percentage of ash in the fuel is 0 032 × 100 = 3 20 per cent At least two estimations should be made, and the average of the results taken.

72 Sulphur in Coal and Coke.—Weigh off 2 gm. of the finely powdered coal Mix with twice the weight of a mixture of two parts of calcined magnesia and one part sodium carbonate (anhydrous) Place the coal and fusion mixture in a platinum crucible, and stir well with a dry glass rod or a stout platinum wire. Fix the crucible in a slanting position on a pipe-clay triangle, and heat over a burner, so that the bottom part of the crucible is brought very gradually to dull red heat Continue the ignition for about an hour Allow to cool, and pour hot water on the mass Wash the whole into a beaker, and add a little bromine water Boil, filter, and wash well with hot water Acidulate the filtrate with hydrochloric acid, boil to drive off all the bromine (until the liquid loses colour), and precipitate with hot barium chloride solution Allow the liquid to stand for 30 minutes, pour off the clear liquid through a filter, boil with fresh hot water (about 80 or 100 c.c), stir up the precipitate, and filter off the liquid again Repeat this three times, then transfer the precipitate to the filter, and wash until free from acid Allow the filter to drain, transfer to a clock glass, dry on the water bath, finishing off in the water oven, and ignite the precipitate as follows. Clean, dry, and weigh a porcelain crucible. Brush off the dried precipitate into this. Take the filter paper, and roll up into a loose ball; coil around this a piece of stout platinum wire Place the crucible on the bench, hold the ball of filter paper over the crucible, and ignite in the flame of a Bunsen burner (without the rose), held in a horizontal position Heat until the paper is consumed, shake off the ash into the crucible, add one drop of strong sulphuric acid on the ash of the paper, and ignite strongly over a Bunsen burner or in the muffle furnace. Cool under the desiccator, and weigh. Ignite again for ten minutes; cool, and weigh again One part of barium sulphate = 0·13734 parts sulphur.

73. Gasification Value of Coal.—It is sometimes required to know what volume of gas, coke, tar, ammonical liquor, etc, can be obtained from a sample of coal. This may be ascertained as follows:—

The moisture, sulphur, and ash having been estimated as already described, prepare a fresh working sample by well mixing the roughly crushed coal, and selecting a portion, as directed, by quartering. Break this up into pieces about the size of a pea Ten grammes of the coal forms a convenient quantity for the test

Take a length of combustion tubing, and pack the coal in loosely, having sealed one end of the tube. The other end is drawn out somewhat, and the end turned down so as to dip into the neck of a small flask immersed in a beaker of cold water. This flask will contain, after the test, the tar and ammonical liquor yielded by the sample of coal From the flask a delivery tube is taken to the top of a large aspirator, provided with a water gauge, to regulate the pressure of the gas coming from the combustion tube The aspirator should be fitted with a run-off cock at the bottom

The heat necessary to gasify the coal is preferably supplied by means of a combustion furnace If this is not available, a row of Bunsen burners, arranged at regular intervals along the length of the tube, will suffice for the purposes of a rough test.

The coal having been carefully packed into the combustion tube, arrange the burners for heating; make all connections with the aspirator, and fill this with water When all is ready light the burner nearest the condensing flask, and commence heating up the tube very gradually. The gas will immediately come off, and one of the bulbs of the gauge will be depressed Now open the run-off tap of the aspirator, and run out sufficient water to restore the level of the two bulbs of the gauge The water must be drawn off very slowly, drop by drop, and the heating must be so regulated that this can easily be done. After a time the other burners may be started, keeping the flames down very low at the commencement, so as to secure a slow and uniform evolution of gas, and a proper condensation of the tar and water, which will condense in the cooled flask. Continue the heating until the emission of gas ceases, measuring the water as it is collected When the gasification is complete, disconnect the apparatus, weigh the tube containing the residual coke, also the flask containing the tar and water, and record the volume of water taken from the aspirator The figures thus obtained will show the volume of gas which may be obtained from the coal, with the quantities of tar and ammonia water, and the percentage of coke which would result from the carbonising.

The test as described here is, of course, only designed to give approximate results By its use, however, a fairly accurate estimate may be obtained of the gasification value of a sample of coal.

74. The Calorific Value of Fuels—The calorific value of fuels is ascertained by means of an instrument called a calorimeter The apparatus here illustrated, which is supplied by Messrs Baird and Tatlock, of London, is that known as Dr. Lewis Thompson's solid-fuel calorimeter

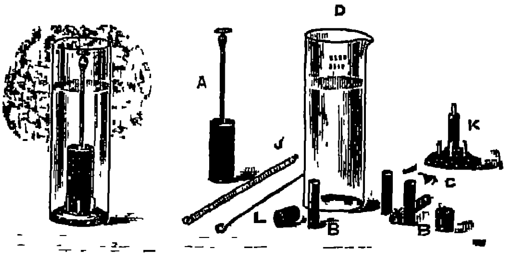


FIG. 25—DR. LEWIS THOMPSON'S CALORIMETER.

The principle of the instrument, briefly stated, is as follows: A weighted quantity of the fuel is mixed with oxygen-yielding substances such as nitrate and chlorate of potash The mixture is then placed in the apparatus and ignited, the products of combustion being passed through a measured volume of water. The rise in temperature of the water during the operation indicates the calorific value of the fuel under examination.

For the purpose of the test, the finely-powdered coal, prepared as already described, is taken Two grammes are weighed out and mixed with 20 grammes of a mixture of potassium chlorate and potassium nitrate in equal proportions. The mixture of coal and potassium salts is then introduced into a cylindrical vessel of copper, called a "furnace," and this furnace is placed in the socket of a metal plate, which is fitted with springs capable of clutching the bottom of a cylindrical copper bell, from the upper part of which extends a long tube, closed by a cock When the powder has been packed into the furnace, a piece of fuse, made by soaking cotton wick in nitre solution, is inserted and lighted The bell is now forced down upon the springs of the base plate, and the whole is immersed in the cylinder, which contains 2,000 c.c. of water at 15·5° C Combustion ensues Allow the combustion to be completed, leave the apparatus immersed in the water for a little time, and then open the stopcock and admit water into the apparatus. Work the calorimeter up and down in the water several times, and observe the temperature of the water with a delicate thermometer

The calorific value of the fuel tested is then calculated from the rise in temperature of the 2,000 grammes of water. For example —

Weight of fuel taken . . .	2 gm.
Temperature of water before combustion .	15 5°
Temperature of water after combustion	20 8°
Rise in temperature .	5 3°
Heat absorbed by apparatus .	10 per cent.

Then, 5 30 + 0·53 = 5·83 × 1,000 = 5,830 = calorific value of fuel.

The heat absorbed by the apparatus is taken as being 10 per cent The calorific value of a fuel as obtained by this apparatus serves as a very reliable basis of comparison when considering the relative merits of a number of different samples of fuel When the moisture, coke, volatile matter, and ash are estimated, as already described, they yield very good figures for comparison, but when the actual calorific value of a fuel is ascertained by means of a calorimeter, the problem is considerably simplified, and, where systematic testing of fuels is undertaken, a Thompson's calorimeter is a good investment. The apparatus as described is used by the Government authorities and railway companies for the purposes of comparing the relative commercial value of coals for which they contract, and may safely be commended to the attention of the large user of fuels

LIQUID FUEL

75. Flashpoint.—The flashpoint should be estimated as described in paragraph 28 Oil or other liquid fuel which has a low flashpoint will, of course, require greater care in storing, etc.

76. Calorific Value.—The calorific value of liquid fuel is estimated in the same manner as in the case of solid fuel (see paragraph 74) The liquid, carefully weighed, is mixed with a small quantity, say 2 gm, of dry kaolin, and the estimation carried out in the same way as with a solid fuel. If the combustion is difficult to start, a little powdered coal or gunpowder, the calorific value of which has been carefully ascertained, may be added before ignition, and the correction for this addition must be made when calculating out the result.

76a. Specific Gravity.—This is estimated by means of the Westphal balance (paragraph 18).

CHAPTER IV.

Miscellaneous Chemicals.

SULPHURIC ACID

77. Specific Gravity.—The specific gravity of sulphuric acid is generally ascertained by the use of the hydrometer. (See paragraph 17.) It may be ascertained more accurately by actual weighing, as described in paragraph 19. In this latter case great care must be taken in the weighing operations, and the outside of the specific gravity bottle must be carefully freed from any acid which may have run down the sides.

78. Real Sulphuric Acid.—The percentage of real sulphuric acid in a sample of vitriol is estimated by titrating a measured volume with standard soda solution. Ascertain the specific gravity of the acid by means of the hydrometer, then measure off 10 c.c. with a pipette (this must be carefully done, as it is very easy to draw up the acid into the mouth), and dilute the 10 c.c. to 100 c.c. Pipette off 10 c.c. of this diluted solution, and titrate with normal sodium carbonate solution, using methyl orange as indicator. In making this titration, a rough test should be first made, as follows: Take 10 c.c. of the diluted solution in a conical beaker, and add about 20 c.c. of distilled water, together with two drops of the methyl orange, by means of a short length of glass tubing. Fill up the pinchcock burette (Fig. 22) with the standard soda solution, and adjust to the mark. Holding the beaker under the burette, press the pinchcock, and run in the solution, drop by drop, shaking all the time. As each drop reaches the alkaline solution in the beaker a reddish colour will appear, disappearing immediately on shaking. By using the pinchcock burette, the delivery of the soda solution may be very easily regulated. When the point of neutralisation is almost reached, the reddish colour produced by the drops of soda solution will become less and less pronounced, and a distinctly yellow colour will take its place The addition of the soda must be continued until this colour is permanent, as, until this is the case, the solution in the beaker is still acid. This may easily be seen by testing with a blue litmus paper.

Now note the number of c.c. of standard soda solution which have been used Empty out the contents of the beaker; rinse several times with tap water, fill up the burette with soda solution again, pipette off a second 10 c c of the acid solution, and repeat the titration In this second test, the number of c.c. being known approximately, the soda solution may be steadily added, with constant shaking, until the point reached in the rough test is reached within one or two c c Now complete the addition drop by drop, and watch very closely for the change of colour. When this is reached, note down the number of c.c of soda consumed, and repeat the titration a third time. The two last titrations will generally agree very closely, if there is a slight difference, the mean of the two readings may be taken.

The calculation of the percentage of real sulphuric acid from the figures obtained as above is as follows —

$$\frac{4\,9 \times \text{c c. soda solution taken}}{\text{specific gravity}} = \%\ \text{real sulphuric acid.}$$

79. Lead.—Dilute a little of the acid with water Add a solution of potassium chromate If lead is present a bright yellow precipitate of lead chromate will be formed.

80 Iron.—Take a little of the strong acid and oxidise with nitric acid Dilute with distilled water, and cool. Now add potassium sulphocyanide solution If iron is present in the acid a red colour will at once result

81. Arsenic.—Take 10 c c of the strong acid, dilute it with water, and pass a current of sulphuretted hydrogen gas through the solution If arsenic is present, a yellow precipitate or sulphide of arsenic will at once be thrown down

Arsenic is present—sometimes in considerable proportions—in sulphuric acid which has been made from pyrites, and has not been purified In acid made from brimstone it is seldom found, and the same may be said of acid made from the spent oxide of the gasworks. The great bulk of vitriol used in manufacturing operations, however, is pyrites acid, and unless it has been purified by treatment with sulphuretted hydrogen (which is the usual process employed for this purpose) it will contain arsenic in more or less degree If arsenic-free acid is required, this should be distinctly stipulated in contracting, and the deliveries should be kept under control by constant testing The detection of arsenic in sulphuric acid is a very simple operation, and can be quickly carried out. If sulphuretted hydrogen is not readily available, then ammonium sulphide solution may be used. This reagent will give the same characteristic yellow precipitate if arsenic be present.

82. Sulphuretted Hydrogen Apparatus.—A handy and easily-prepared apparatus for the generation of sulphuretted hydrogen gas for the above and other similar tests where this gas is employed, is made by taking a good, large aspirator of about 2 litres, with a tubulure at the bottom, and with one neck Fit a rubber stopper to the tubulure so as to close it tightly. Into the neck fit a rubber stopper, pierced with two holes Through one of the holes a piece of glass tubing, bent at right angles, is fitted, and connected by means of a rubber joint to the delivery tube for the gas Into the other hole a thistle funnel is fitted, reaching down beneath the surface of the liquid in the apparatus when in use. When the gas is required, a charge of sulphide of iron, broken up into small pieces, is placed in the vessel; water is added to the depth of about two or three inches, and the thistle safety funnel adjusted so that the end is sealed by the water Now run a little strong acid down the funnel, give the apparatus a shake, and in a few seconds the current of gas will begin to be evolved. Insert the end of the delivery tube in the solution to be tested, and allow the gas to bubble through. If arsenic is present, a yellow precipitate will immediately fall After use, the sulphuretted hydrogen apparatus should be washed out by filling with water through the neck, and running it out through the tubulure at the bottom Repeat this washing several times to remove the acid, and leave two or three inches of water in the apparatus, which is now ready for use again.

83. Nitrous Acid—Make a solution of four grammes of pure diphenylamine in pure, strong sulphuric acid, which must be free from nitrogen compounds; to ensure this, boil the acid with a little sulphate of ammonia. To 100 c.c acid add 10 c.c.

water, and in this solution dissolve the diphenylamine Place 2 c c of the acid to be tested in a test tube. Down the side of the tube allow 1 c c. of the diphenylamine solution to run, so that the two liquids form two separate layers. If nitrogen compounds are present, a bright blue colour will appear where the two liquids meet. If dilute acid is being tested, reverse the order of mixing

BLEACHING POWDER

84. In many industries where large quantities of bleaching powder (and bleaching liquor) are constantly used—and particularly in the paper trade, the bleaching and dyeing trades, and many others—it is most important that a constant check should be kept on the strength of the deliveries. In some mills, however, very little testing is done of the supplies of bleaching materials, with the inevitable consequence that the articles are seldom up to the proper strength, with a corresponding loss of economy in working.

Bleaching powder is generally sold as containing from 35 per cent to 36 per cent of "available chlorine," and this is the constituent which should be estimated in testing the material. This point is not infrequently lost sight of by consumers of bleaching powder, who often have an impression that the strength of the powder is indicated by the specific gravity of the solution obtained from it. This is a very erroneous idea. The specific gravity of a solution made up from bleaching powder may be greatly affected by the admixture of some useless soluble substance such as common salt, which has no bleaching value, but which heightens the specific gravity of the solution. The Twaddell hydrometer may be safely used in testing the strength of the working bleach liquor, *after* the bleaching powder itself has passed the chemical test here described; but for the purpose of estimating the strength of the powder itself, the use of the hydrometer is unreliable.

85. Sampling Bleaching Powder.—When a delivery of bleaching powder is received, it should be sampled at once. This precaution is especially necessary in warm weather, when the strength of bleach rapidly drops in the course of a few days. For instance, a delivery which tested 36 per cent available chlorine when received, after two days indicated only 35¼ per cent, and after a further two days the figure was 34½ per cent, while after the lapse of a week the strength of the consignment was only a little over 33 per cent This was in summer, but the *immediate* sampling of deliveries of bleach is a matter which should not be neglected.

Assuming that thirty casks of bleach have been received, it is a good plan to sample alternate casks These are generally numbered consecutively, and a note should be made of the numbers, and any other marks, such as the brand, with the date of delivery and the date when sampled If only a few casks are to be tested, say ten or a dozen, it is far safer to sample each one; the sample thus obtained is much more reliable than when, as is done in some mills, only three or four casks are taken, no matter how large the consignment which they are supposed to represent.

Taking the instance mentioned of a thirty-cask delivery, take the numbers of marks, as indicated, of each cask in the parcel. Then proceed to sample each alternate cask For this purpose a large auger is necessary, and also a brace with a bit of a suitable size. The casks should be on their sides, in such a position that the ends are readily accessible, and the sampling should be done from the end After boring a hole in the head of the cask with the brace and bit, clear away the splinters of wood, insert the auger, and work it in up to the handle, twist it round once or twice, withdraw the instrument, and empty the contents on a sheet of stout brown paper spread out on the floor. Repeat this operation with each of the casks which are to be sampled, taking care to penetrate as far as possible into each one. Having sampled the whole of the casks, well mix the heap of powder thus obtained, throwing out any bits of wood, and crushing any lumps After thoroughly mixing, take three clean and dry sample bottles, each holding about from four to eight ounces, and place them close together on the sheet of paper containing the sample Now take a handful of the bleach, and shake it over the bottles, so that a portion drops into each alternately, the rest, of course, falling on the paper Repeat this until the whole of the pile has thus been re-sampled, and the sample bottles are full, then insert tightly-fitting corks into the bottles, and the sample is ready for testing. If a reference sample is required, seal one of the bottles, and label it with all the particulars—date, brand, number of casks, etc

The points to be observed in sampling a parcel of bleaching powder are · (1) A good number of casks should be taken for the test, (2) the sample should be taken from a point as far down the cask as possible, and for this purpose a long auger is necessary; (3) the sample so taken must be well crushed and thoroughly mixed before the sample bottles are filled, and the sample sifted through the fingers when filling the bottles.

86. Estimation of Available Chlorine in Bleaching Powder.—The apparatus necessary for this test is as follows A white porcelain dish, a 10 c.c burette, with glass stop cock and graduated in tenths of a c c., a Bink's burette holding at least 20 c c. (Fig 23), standard solution of arsenious acid, solution of indigo, glass stirring rod (hooked end), large porcelain mortar and pestle, and a 500 c.c stoppered flask

Weigh off 10 gm of the well-mixed sample of powder, place in the mortar, add a little distilled water, and grind up into a cream Allow to settle for a few seconds, and pour off the liquor through a funnel into the 500 c c flask Now add a further quantity of water, again grind up thoroughly, allow to stand for a few seconds, and again pour off the liquor into the flask Repeat this operation until about 200 or 250 c c of liquor has been transferred from the mortar to the flask, then wash the residue remaining in the mortar through the funnel by means of a wash bottle, thoroughly washing both the mortar and the funnel after the final transference has been made. Remove the funnel and well mix the contents of the flask, then make up to the 500 c c mark, and agitate constantly for half an hour. Now take the Mohr's burette, previously washed out and drained. Again agitate the contents of the 500 c c. flask, pour a little of the liquor into the burette, and rinse out with it; (care must be observed throughout this test to agitate the contents of the flask each time a portion is abstracted, so that the proper amount of sediment is taken, in addition to clear liquor). Fill up the 10 c c. burette with the arsenious acid solution, and run out exactly 10 c c. into the porcelain dish; now add 2 or 3 drops of indigo solution, fill up the Mohr's burette to the mark with the agitated bleach liquor, and add it drop by drop to the arsenious acid in the basin, stirring well during the addition. At each addition of the bleach solution a blue colour will be produced, which, however, will disappear on stirring. Continue this until a brown colour results from the addition of the bleach liquor. The addition of the bleach solution must be continued until this colour remains permanent. Now read off the number of c.c. of bleach liquor which have been used, empty the burette of the bleach liquor, and re-fill. Proceed to make a second test in precisely the same manner as above described. Add the last 2 or 3 c.c. very carefully, and watch closely the end point of the reaction. It is generally advisable to make a third test, and, as a rule, the second and third estimations will give almost identical results. If there is any difference in the figures, the average of the two tests should be taken.

The percentage of available chlorine contained in the sample may now be calculated from the following table:—

10 GM. BLEACH MADE UP TO 500 C.C.

c.c. Bleach used	% Chlorine	c.c. Bleach used	% Chlorine	c.c. Bleach used	% Chlorine.
13·0	38·46	15·0	33·33	17·0	29·41
13·1	38·26	15·1	33·11	17·1	29·23
13·2	37·87	15·2	32·89	17·2	29·06
13·3	37·51	15·3	32·67	17·3	28·90
13·4	37·31	15·4	32·46	17·4	28·73
13·5	37·03	15·5	32·25	17·5	28·57
13·6	36·76	15·6	32·05	17·6	28·46
13·7	36·49	15·7	31·84	17·7	28·24
13·8	36·23	15·8	31·64	17·8	28·08
13·9	35·97	15·9	31·44	17·9	27·93
14·0	35·71	16·0	31·25	18·0	27·77
14·1	35·46	16·1	31·05	18·1	27·62
14·2	35·21	16·2	30·86	18·2	27·47
14·3	34·96	16·3	30·67	18·3	27·32
14·4	34·72	16·4	30·48	18·4	27·17
14·5	34·48	16·5	30·30	18·5	27·02
14·6	34·24	16·6	30·12	18·6	26·88
14·7	34·01	16·7	29·94	18·7	26·73
14·8	33·78	16·8	29·76	18·8	26·59
14·9	33·55	16·9	29·58	18·9	26·45

AVAILABLE CHLORINE IN BLEACHING LIQUOR

87. Measure off 25 c.c. of the sample of liquor into a 250 c.c. flask, and dilute with water to the mark. Mix thoroughly, and fill a 50 c.c. burette with the solution. Fill a 10 c.c. burette with the standard arsenious acid solution (paragraph 53), and measure off 10 c c. carefully into a white porcelain dish, add a few drops of indigo solution (paragraph 65), and into this run the bleach liquor until the blue colour changes to a yellowish brown. Make at least two duplicate tests, and take the average of the two readings of bleach liquor consumed Divide the number of c c. used into 100; the result is the percentage of available chlorine present in the liquor by volume Bleaching liquor is generally sold as containing 7 per cent available chlorine

PYRITES

Preparation of the Sample.—In the testing of pyrites the sample must be carefully prepared, otherwise endless trouble will be caused in the analysis The sample should be broken up roughly in an iron mortar, carefully mixed, and re-sampled by quartering. Reduce the final sample to an impalpable powder by grinding in an agate mortar, operating upon small quantities at a time, and transferring the ground sample to a small bottle as it is reduced The grinding of the sample cannot be carried too far, and, when completed, it should not be possible to feel even minute lumps when a little is taken between the fingers.

88. Sulphur.—Weigh off two lots of ·5 gm. each of the finely powdered sample, and place in conical beakers over the water bath Prepare a mixture of 3 parts of strong nitric acid and 1 part strong hydrochloric acid Place 10 c c. of the mixed acids in the beakers, insert a funnel in each, and heat on the water bath Sulphur may at first separate out; continue the heating until this is dissolved, then remove the funnels, and evaporate to dryness. Add a little strong hydrochloric acid (about 5 c c), and evaporate to dryness again; add a further 5 c c. of hydrochloric acid, and again evaporate; by this time the nitric acid should all have been driven off. Moisten the residue with strong hydrochloric acid, add hot water, filter, and well wash the residue on the filter. The residue on the filter paper is ignited (see paragraph 45) and weighed; it consists of silica and other matters which are classed as "insoluble"

To the filtrate from the insoluble matter add ammonium hydrate in slight excess. This will precipitate the iron. Filter hot, and wash the iron precipitate well with hot water; continue the washing until 1 c.c. of the filtrate shows no milkiness with barium chloride solution. Keep down the filtrate and washings to about 200 c c, and to this add a little strong hydrochloric acid until just on the acid side, and heat the solution Now heat to boiling a solution of barium chloride, and with this precipitate the sulphur as barium sulphate. Allow to stand for about thirty minutes, then pour the clear liquor through a filter; add about 80 to 100 c c *boiling* water to the precipitate in the beaker, boil for a minute, allow to settle, and filter the clear liquid as before Repeat this three times, then wash the precipitate on to the filter, wash well, dry in the water oven, ignite, and weigh. One part barium sulphate = 0 13734 sulphur

88a. Moisture.—Weigh off 10 gm of the finely-ground sample, dry at 105° C, cool, and weigh. The loss multiplied by 10 = per cent moisture

89. Copper.—Weigh off 1 gm of the finely-ground sample Place this in a small beaker, cover with strong nitric acid, and heat on the water bath to dryness. Add a little strong pure sulphuric acid, and place on the sand bath, heating until all acid fumes have been driven off. Cool, add water, and boil When cool again, add methylated spirit (about a third of the volume of water in the beaker or basin), allow to stand all night, and filter off in the morning Wash the residue on the filter with a mixture of methylated spirit and water (one of spirit to two of water), until a drop of the filtrate ceases to show a blue colour on the addition of nitric acid Pass sulphuretted hydrogen through the filtrate and washings (see paragraph 82), and allow the precipitate so obtained to stand for several hours. Filter, and wash with water through which sulphuretted hydrogen gas has been passed, and containing also a little pure sulphuric acid Dry the filter in the water oven Place the dried precipitate, with filter paper, in a Rose's crucible (Fig. 24), into which a current of coal gas is passed, add a little pure sulphur, and ignite. Cool in the desiccator and weigh When testing Spanish pyrites, according to Lunge and Hurter's "Alkali Makers' Pocket Book," 0·0005 gm., together with the weight of the filter ash, must be deducted from the sulphide of copper thus obtained, as a correction for the antimony and bismuth present in the ore. The percentage of copper is then calculated from the figures obtained from 1 gm. of the sample. One part Cu_2O = 0·79874 of copper

BURNT PYRITES

90. Sulphur.—Sulphur in burnt pyrites is estimated in exactly the same manner as described in paragraph 88.

91. Copper—Proceed as in paragraph 89, dissolving the sample in hydrochloric acid, with the addition of a little nitric acid The same correction for antimony and bismuth is made when these metals are present in the ore.

92. Moisture.—See paragraph 88*a*.

CAUSTIC SODA

93 Sampling.—The sample should be taken from underneath the surface of the contents of the drum to be sampled. If a delivery of, say, 20 drums is to be tested, open every third drum, scrape away the top layer, and draw a fair sample. Repeat this with each drum selected for testing Mix the sample well, and reduce to a bulk which will fill a fairly large sample bottle with a wide mouth. Tightly stopper the bottle, and, if the test is not to be made at once, seal with wax When weighing out quantities for testing, avoid all pieces on which a crust of carbonate has formed, if necessary, scrape off this crust before weighing.

94. Available Alkali.—Weigh off 25 gm of the sample, dissolve in water in a beaker over the water bath, and make up to 500 c c. Pipette off 20 c c. (= 1·0 gm of sample) into a beaker, dilute with water, add a few drops of methyl orange solution, and run in normal hydrochloric acid until the red colour is permanent. Each c.c. of acid used = 0·031 gm. Na_2O

95. Insoluble—Weigh off 10 gm of the sample; dissolve in a beaker over the water bath; filter, wash the insoluble residue on the paper, dry the paper and contents on a clock glass over the water bath, and place in the water oven until quite dry Clean and weigh a porcelain crucible or a platinum dish, place the dried filter paper in this, and ignite over a Bunsen burner or in the muffle furnace (paragraph 45). Cool under the desiccator, and weigh. The residue multiplied by 10 gives the percentage of insoluble matter present.

CAUSTIC LIQUOR.

96. Available Alkali.—Weigh off into a beaker 15·5 gm. of the liquor, make up to 500 c.c., mix well, pipette off 50 c.c.

into a beaker, and titrate with normal hydrochloric acid and methyl orange (See paragraph 94) Each c.c. of normal acid used = 2 per cent of available alkali. The specific gravity should be carefully taken, as caustic liquor is usually sold as so many degrees Twaddell

SALT CAKE.

97. Sodium Chloride.—Well mix the sample, grind up in a mortar, and again mix. Weigh off 0·585 gm of the prepared sample, and dissolve in distilled water Neutralise by using pure nitric acid and pure carbonate of magnesia, and titrate with the deci-normal solution of silver nitrate until the reddish colour remains on stirring Each c c. of silver solution = 1 per cent of sodium chloride present in the sample; this should be under 1 per cent

98. Free Sulphuric Acid.—Weigh off 4·9 gm of the prepared sample (see above), and dissolve in a beaker, using distilled water Colour the solution with litmus, and titrate with the normal sodium hydrate solution. Each c c. of sodium hydrate used is equal to 1 per cent of free acid

99 Insoluble Matter—Weigh off 10 gm of the prepared sample (see above) into a beaker, and dissolve in distilled water. Filter through a *small* filter paper, well wash the insoluble residue on the paper, and dry, first on a clock glass over the water bath, and then in the water oven Clean and weigh a porcelain crucible, and ignite the paper and residue as in paragraph 45. Cool under the desiccator, and weigh The residue multiplied by 10 gives the percentage of insoluble matter present.

100 Iron.—Dissolve a quantity of the sample in water in a beaker, adding a few drops of pure nitric acid free from iron. Cool, and add a solution of potassium sulphocyanide. A red colour indicates the presence of iron.

101. Sodium Sulphate—(1.) Dissolve 1 gm. of sample in water, adding a little hydrochloric acid to complete solution, if necessary Add ammonium chloride, ammonium hydrate, and ammonium oxalate. Filter. To the filtrate add a little pure sulphuric acid, evaporate to dryness, and ignite. Add a small piece of solid ammonium carbonate, ignite again, cool, and weigh the residue, which, multiply by 100, and call this figure A

(2) Dissolve 10 gm. as above, precipitate with ammonium chloride, ammonium hydrate, and ammonium oxalate, and filter To the filtrate add ammonium phosphate solution, settle for about twenty-four hours, filter, wash with ammonia water, dry the precipitate, and weigh the magnesia as pyrophosphate. Weight × 10×0·36036 = per cent. MgO

(3) Multiply per cent. sodium chloride (paragraph 97) by 1·2136, and call this B Multiply per cent. MgO (see above) by 3, and call this figure C Add together B and C, and deduct from A The result is per cent. sulphate of soda by direct estimation.

WASHING SODA (SODA CRYSTALS)

102. Insoluble Matter—Crush the crystals roughly in a mortar, and weigh off 10 gm of the sample. Dissolve, filter, wash free from alkaline reaction, ignite, and weigh as in paragraph 45a. The residue multiplied by 10 gives the percentage of insoluble matter present If this is very small, 20 gm. may be weighed off for the test, and the residue multiplied by five

103 Available Alkali—Weigh off 15 5 gm of the sample, dissolve in a beaker with distilled water, and pour the solution into a 500 c c. flask, washing the beaker well into the flask, and make up to the mark. Titrate 50 c c of this solution with normal sulphuric acid, using methyl orange as indicator. (See paragraph 78) Each c c. of acid taken = 2 per cent. of Na_2O.

SODA ASH.

104. Sampling.—Sample at least every third cask or bag in the delivery. If only a small consignment is to be tested, each package should be included in the sample. If in casks, sample as in the case of bleaching powder (see paragraph 85); if in bags, discard the top layer, and take a sample from below the surface. Reduce the sampling by quartering. Grind the working sample in a mortar, and well mix.

105 Insoluble Matter.—Weigh off 10 gm. of the prepared sample, and dissolve in a beaker over the [illegible] constantly Allow the sample ample time for solution, then filter through a small filter paper; wash the insoluble residue on to the filter, and wash well with hot water Dry the filter and residue, first on a clock glass over the water bath, and then in the water oven Clean and weigh a porcelain or platinum crucible, and in this ignite the filter paper and residue over a Bunsen burner or in the muffle furnace (See paragraph 45) Cool in the desiccator, and weigh Deduct the ash of the filter paper, and multiply the result by 10, which will give the percentage of insoluble matter

106. Available Alkali.—Weigh off 15·5 gm of the prepared sample (see above), and dissolve in a beaker over the water bath, using distilled water, and stirring constantly When dissolved, pour into a 500 c.c stoppered flask, cool, and make up to the mark when cold. Mix well, and pipette off 50 c c. of the solution into a conical flask; add a few drops of methyl orange, and titrate with normal sulphuric acid. Each c.c of acid used = 2·0 per cent of Na_2O.

BRIMSTONE.

107. Moisture.—Crush the sample roughly, and keep in a sample bottle, tightly corked Weigh off 10 gm. and dry at 100° C in the water oven until the weight is constant. Cool under the desiccator, and weigh. The loss multiplied by 10 gives the percentage of moisture. If this is very low, a check test may be made on 50 or 100 gm. of the sample.

108. Ash.—Weigh off 10 gm of the sample in a porcelain dish, previously weighed. Heat the dish over a Bunsen burner or in the muffle furnace until the sulphur is completely burnt off. Cool under the desiccator, and weigh. Ignite again, and repeat until the weight is constant The residue multiplied by 10 gives the percentage of ash.

109. Sulphur.—Weigh off 2·5 gm. of the dried sample, after the estimation of the moisture. Extract this as described in the case of spent oxide (paragraph 148). The sulphur residue multiplied by 40 gives the percentage of sulphur on the dried sample.

SALT (COMMON SALT AND ROCK SALT)

110. Moisture.—Weigh off a clean platinum crucible, into which place 10 gm of the sample, well ground and mixed. Ignite gradually over a Bunsen burner or in the muffle furnace to a dull red heat The loss multiplied by 10 gives percentage of moisture.

111. Insoluble Matter.—Dissolve 10 gm. of the salt in distilled water; filter, wash well, and dry the filter paper in the water oven. Weigh off a clean platinum or porcelain crucible, and in this ignite the filter paper and residue Deduct the weight of the filter ash, and multiply the residue by 10, which gives the percentage of insoluble matter

112 Chlorine—Weigh off 5·85 gm. of the sample as received Dissolve in distilled water and make up to 500 c.c. Pipette off 25 c c, neutralise the solution, and add a few drops of potassium chromate, so as to produce a distinctly yellow colour. Run in the deci-normal silver solution until the precipitate is of a faint pink tinge. Deduct 0·2 c.c from the number of c c. of silver solution required Multiply the figure so obtained by 2, the result is the percentage of sodium chloride contained in the sample

NITRATE OF SODA

113. Moisture.—Well grind and mix the sample, keeping in a well-corked sample bottle. Clean and weigh a platinum or porcelain crucible or dish, and dry 10 gm in the hot-air oven at 130° C. until the weight is constant The loss multiplied by 10 gives the percentage of moisture

114. Insoluble Matter.—Dissolve 10 gm. in water, filter, well wash the residue on the filter paper, and dry in the water oven. Weigh a platinum or porcelain crucible, and ignite the filter paper Cool under the desiccator and weigh. The residue multiplied by 10 gives the percentage of insoluble matter.

115. Sodium Chloride.—Weigh off 5·85 gm., and estimate the sodium chloride as in paragraph 112.

116 Sulphate of Soda.—Weigh off 20 gm. of the sample, and dissolve in water. Acidify with a little pure hydrochloric acid, and heat to boiling. Precipitate with a solution of barium chloride; filter, wash and weigh the barium sulphate. (See paragraph 88) Multiply the weight of barium sulphate obtained by 5, and then by 0·60944 The result is the percentage of sulphate of soda present

117. Nitrate of Soda.—Add together the moisture, insoluble matter, sodium chloride, and sodium sulphate, and deduct from 100; the result gives the percentage of nitrate of soda by difference

LIMESTONE

118 Insoluble Matter.—Grind up the sample well in a mortar, and mix thoroughly Weigh off 1 gm into a beaker, and dissolve it in pure hydrochloric acid When dissolved, add water, filter, and well wash the residue on the filter paper Dry the paper in the water oven, and weigh until constant. Clean and weigh a platinum or porcelain crucible, and ignite the weighed paper and residue The residue after ignition, multiplied by 100, is the total insoluble matter. The difference between the weight after drying and the weight after ignition, multiplied by 100, is the percentage of organic matter

119. Lime.—Weigh off 1 gm. of sample, and dissolve in 25 c.c normal hydrochloric acid. Add a few drops of methyl orange, and titrate with standard alkali Deduct the number of c c. alkali consumed from 25, and multiply the result by 5. This gives the percentage of calcium carbonate present

120 Iron.—Dissolve about 2 gm of the sample in hydrochloric acid Add a little strong nitric acid, and a little potassium sulphocyanide solution A blue colour shows that iron is present

CHLORIDE OF POTASH.

121. Moisture.—Grind and mix the sample well Weigh off 10 gm into a platinum or porcelain crucible, and heat over a Bunsen burner or in the muffle furnace to redness. Cool under the desiccator and weigh. The loss multiplied by 10 gives the percentage of moisture.

122. Potassium Chloride.—Weigh off accurately 0·746 gm of the prepared sample. Dissolve in water, neutralise, and titrate with deci-normal silver solution The number of c.c. of silver solution gives the percentage of potassium chloride contained in the sample

SULPHATE OF POTASH.

123. Free Acid—Estimate as in the case of salt cake (see paragraph 98), taking 4·9 gm of sample

124 Potassium Chloride.—Estimate as in chloride of potash (see paragraph 122), taking 0·746 gm. of sample.

125. Insoluble Matter.—Estimate as in salt cake (see paragraph 99), taking 10·00 gm. of sample

126. Iron.—This may be detected as in salt cake (paragraph 100).

CHLORATE OF POTASH

127. Chloride—Weigh off 50 gm of the sample (well ground and mixed). Dissolve in *distilled* water, neturalise, and titrate with deci-normal silver solution. Each c c of silver = 0·015 per cent. of potassium chloride. For insoluble matter, iron, etc, see sulphate of potash

CHAPTER V.

Oil Testing.

FLASH POINT

128. The Abel Flash-Point Apparatus.—The apparatus here illustrated is the Abel standard flash-point apparatus This is constructed to the requirements of the Board of Trade, and is supplied by Messrs. Baird and Tatlock, of London, at the price of 5 guineas, with Board of Trade verification Where liquid fuel is used, and for all purposes of testing illuminating oils the Abel flash-point apparatus is infinitely the best The figures obtained by it are thoroughly reliable; the tests are conducted under precisely the same conditions, and therefore the results, as a whole, are comparable. This is a very important point, as, in some of the cheaper and more imperfect types of flash-point apparatus, it is quite possible, by a little manipulation, to obtain any desired result; therefore, although the Abel apparatus is rather an expensive one, it is here recommended for the reasons above given.

The apparatus, when in use, must be placed in a position where it is not exposed to currents of air or draughts. Fill the water bath by pouring water into the funnel, until it runs out from the spout at the other side. Then, by means of the lamp underneath the apparatus, heat up the water bath until the thermometer on the left shows 130° F This temperature is best attained by heating water in a separate vessel until it approaches about 120° F., the water bath should then be filled with this hot water, and the temperature of the jacket raised by means of the lamp to 130 degrees. Now open the cup in the centre of the apparatus, and pour in the oil to be tested, until the level of the liquid just reaches the point of the gauge which is fixed in the cup. In warm weather the temperature of the room where the samples have been kept should be noted If this exceeds

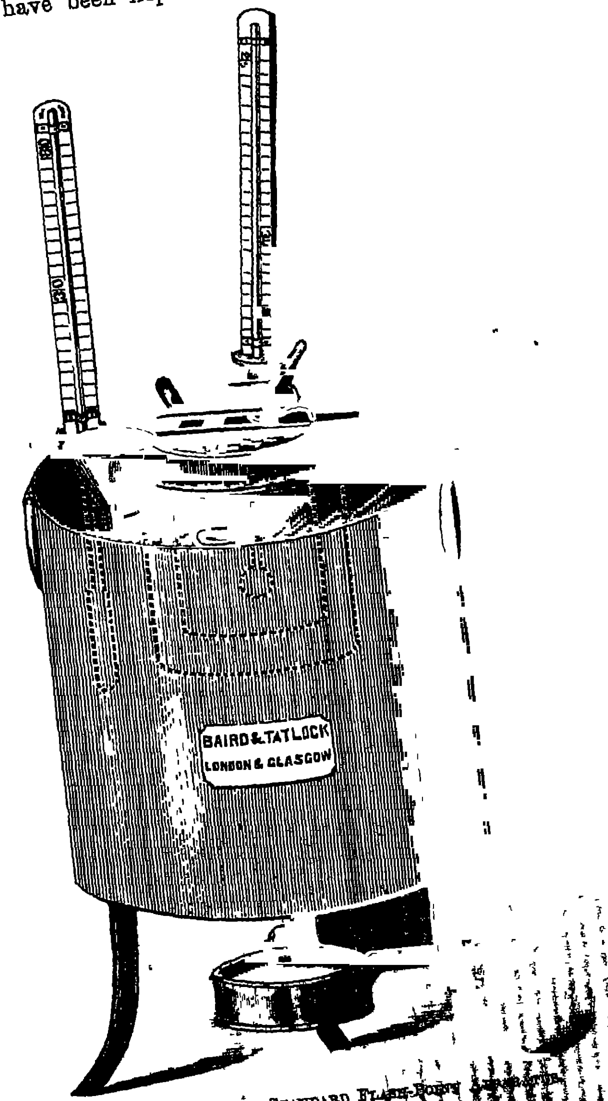


FIG. 26.—THE ABEL STANDARD FLASH-POINT APPARATUS.

65° F., cool the samples down to about [illegible] immersing the bottles containing them in cold water.

The test cup having been filled to the proper height, the lid, with the slide closed, is now put on, and the cup is placed in the bath. Adjust the thermometer in the lid of the cup, so that the bulb is just immersed in the oil, this position must not, under any circumstances, be afterwards altered If the cup is now in its proper position, the scale of the thermometer will face the operator. Now light up the small test jet fitted to the slide of the cup (a gas supply having already been brought to this test jet by means of a rubber tube), hang up the pendulum supplied with the apparatus from a nail fixed so that the pendulum swings in front of the operator, and prepare for the actual test. When the temperature, as indicated by the thermometer in the oil cup, has reached about 70° F the operation of testing may be commenced, the test flame being applied as the temperature rises, in the following manner The slide is slowly drawn open, while the pendulum performs three oscillations, and is closed during the fourth oscillation

Raise the temperature of the oil cup to 70° F., and apply the test flame. Watch for a very slight flash to pass over the surface of the oil in the cup when the slide is drawn If no flash appears, close the slide at once, and allow the temperature to rise two or three degrees. Now apply the flame again, and watch for the flash. If this does not appear, allow the temperature to rise again two or three degrees, and once more test In this way, at, say, 80° F., when the slide is drawn, the operator will notice a slight flame pass over the surface of the oil when the test jet is applied. The flame may be very slight, and the cup must be closely watched during the test, or the flame may be overlooked altogether. A little practice, however, will enable the operator to detect a very faint flash in the cup. Assuming that the flash point thus roughly ascertained is 80° F, as registered by the thermometer in the cup, empty the cup altogether, turn down the lamp under the water bath, and allow the temperature once more to sink Fill the cup again, as already described, taking care that the gauge is not passed, and once more raise the temperature as before, testing now at each degree When the thermometer indicates 78°, the flash may be observed. If this is so, a third test should be made, once more emptying out the oil from the cup, allowing the temperature of the water bath to fall, replenishing the test cup with a fresh portion of the sample, and again raising the temperature very gradually, testing at each degree. This third operation will, in all probability, confirm the second test, and therefore the flash point of the oil under examination is 78° F

129. The Abel-Pensky flash-point apparatus is similar to the Abel apparatus already described, with the addition of clockwork mechanism to open the cover and depress the test flame. This form of flash-point apparatus is also supplied by Messrs. Baird and Tatlock, at the price of £6.

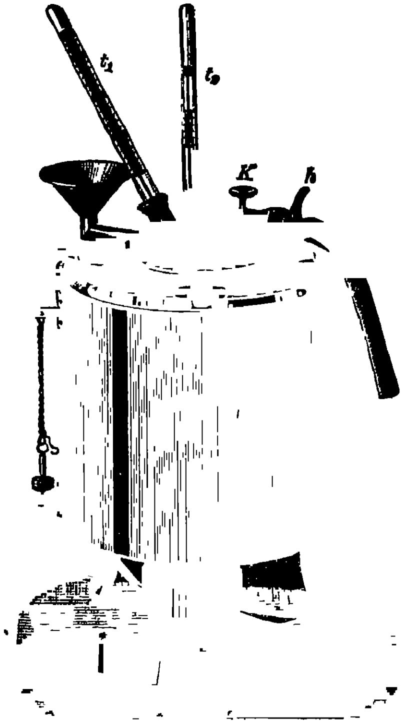


FIG 27 —ABEL-PENSKY FLASH-POINT APPARATUS

130. Gray's Flash-Point Apparatus.—For estimating the flash point of lubricating oils, a reliable apparatus is Gray's, which is arranged to test up to 550° F. It is supplied by Messrs

Baird and Tatlock at the price of £3 15s., exclusive of thermometers, which, ranging from 100° to 550° F., cost 6s. each.

The apparatus consists of a brass oil cup of the same dimensions as the oil container used in Government standard petroleum testers (Abel's). On this cup, which is set on a suitable stand, is placed a tight-fitting cap or lid. Through the lid a steel shaft passes, carrying on the top a small bevelled wheel, the bevel of which is milled, and on the lower part two sets of stirrers, one of which is below the surface of the oil, and the other in the vapour space above. A horizontal shaft carried on two standards terminates at one end in a bevelled wheel, which gears with the wheel first mentioned, and at the other end in a disc, the outer rim of which is made of bone or other suitable non-conductor of heat, and on this disc is fixed a handle for rotating the shaft.

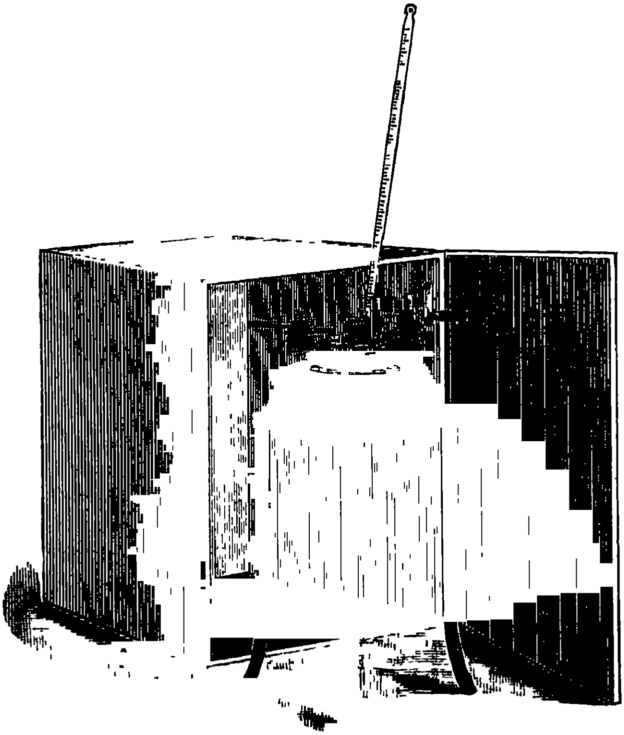


FIG. 28.—GRAY'S FLASH-POINT APPARATUS.

This horizontal shaft carries a collar, from which there projects two small pins at diametrically opposite points. By sliding the shaft slightly to the right, the bevelled wheels are drawn out of gear, and the pins projecting from the collar put in position for actuating the testing arrangement On the lid proper there are three orifices, one immediately in front of the test light, and the other two at either side of it. Above the lid is the sliding cover, in which there are two orifices corresponding to those on the fixed lid. When the bevelled wheels are in gear the sliding cover is held in its normal position by a spring, and the orifices in the fixed lid are covered. When the sliding cover is moved by bringing the pins in contact, and turning the disc about a quarter of a turn round, the orifices in it coincide with those on the fixed lid, and at the same time the test light is depressed, and brought into position for producing the flash when the proper temperature has been reached In places where gas is not available, a good substitute to use for the test flame may be obtained by passing a current of air or hydrogen through cotton wool or other convenient medium saturated with gasoline or other light hydro-carbon spirit.

The apparatus is used as follows. Fill the oil cup up to the inside mark with the oil to be tested, replace the cap, and insert the thermometer in the tube provided for it. Apply a light to the heating lamp, the heat from which is applied through the medium of a convenient sand bath. Light the test jet also, and adjust the flame to about ⅛th of an inch in length During the heating of the oil, the stirrers should be moved at short intervals by turning the handles several times. The more rapidly the oil is heated, the oftener the stirrers should be rotated When nearing the point at which the oil is expected to flash, it is advisable to reduce the rate of heating, in order that the observations may be more readily and accurately made If necessary, a rough test may be performed first, to give some indication of the temperature at which the oil is likely to flash Observations may be taken every degree or half degree, a few turns of the stirrer being given between each test. The rate of heating is immaterial, provided it is not too rapid to allow of readings being taken.

131. Simple Flash-Point Test.—The following is a simple method of ascertaining the flash point of an oil, when a more elaborate apparatus is not available. Where accuracy is required, such a test is, of course, of little value, and the standard flash-point apparatus should be used, but as a rough test the one here indicated is often used:—

Take a small basin, preferably a copper dish, holding about 25 c.c. Fix a tripod stand over a Bunsen burner, and on this place a sand bath, made by filling a copper, tin, or iron dish with sand. Place the copper dish in the bath, sinking it well in the sand. The oil dish should have a mark punched on the side, and in each test the oil should reach this level. A small gas jet is also required, with which to ignite the vapour when the flash point is reached. For this purpose a mouth blow-pipe is often used. To the blow-pipe a gas supply is brought by a rubber tube attached to the mouth-piece end, and when the gas is ignited a small flame, about the size of a pea, is easily obtained. Having fixed up the apparatus, the test is made as follows. Fill the oil basin to the level, and hang a thermometer so that the bulb is immersed in the oil. Light the burner under the sand bath, and raise the temperature slowly—about 10° F per minute is the usual rate. As the temperature of the oil rises, apply the small blow-pipe jet at each rise of two degrees. When the flash point is reached, a blue flame will shoot across the surface of the oil. The test should be repeated, and three separate readings may be taken with advantage.

The above test gives what is known as the "open" flash point; the standard instruments give the "close" flash point. The close test is always lower than the open test.

132. A Safe Flash Point.—For lubricating purposes, within certain limits, it may safely be taken as a rule that the higher the flash point the safer is the oil. For general purposes, a lubricating oil should not flash under 330° F., while for mills, where inflammable matter is plentiful—cotton mills, for example—a safe limit is about 350°. In the case of cylinder oils, the flash point is generally required to be not less than 500° F.

VISCOSITY.

133. Pipette Method.—An approximate and simple method of comparing the viscosity of different samples of oil is as follows: A pipette, say of 50 c.c. capacity, is taken, and two marks made with a file, one on the upper part of the stem, and the other on the lower part. A piece of rubber tubing is slipped over the mouth of the pipette, and a spring clip fixed on this rubber tube. The pipette is filled to the upper mark by suction, releasing the clip, while the oil is being drawn up to the mark. The pipette is now fixed in the stand by means of a clip, a vessel being placed beneath to receive the oil. The clip is released, and the number of seconds occupied by the oil in flowing from one mark to the other is ascertained. This operation is repeated, and the average number of seconds is taken as being an approximate viscosity, for purposes of comparison. The pipette should then be cleaned with ether, dried, and the same experiment repeated with pure rape oil. In such a rough test it is important that all the samples tested should be used at the same temperature. For this purpose vessels of each oil to be tested should be kept placed side by side in the same room—carefully marked, of course. The pipette is then filled with each oil in turn, and the number of seconds occupied in each case is observed, as compared with the standard rape oil. If proper precautions are taken to ensure uniformity of temperature in each sample, and if several tests are made of each oil under examination, this test, although rough, is yet a useful guide for the approximate comparison of a number of samples.

The Viscometer.—For the purpose of accurately estimating the viscosity of oils, a properly constructed viscometer is necessary. The standard instrument for testing the viscosity of oils is Redwood's Viscometer. (Figs. 30 and 31.) This apparatus is made and supplied by Messrs. Baird and Tatlock, of London, at the price of £4 10s.

134. Redwood's Viscometer.—The instrument consists of a silvered-brass oil cylinder, furnished with an agate jet, and surrounded by a copper bath. A copper tube, closed at the lower end, projecting at an angle of 45° from the side of the bath near the bottom, provides a means of heating the liquid contained in the bath, and, by the use of a revolving agitator, which forms part of the apparatus, the heated liquid rising from the copper tube can be uniformly distributed through the bath.

The agitator carries a thermometer to indicate the temperature of the bath The oil cylinder is furnished with a stopper, consisting of a small brass sphere attached to a wire, the sphere resting in a hemispherical cavity in the agate jet. A short standard, attached to the oil cylinder, carries a clip to support a thermometer in the oil. Inside the oil cylinder, and at a short distance from the top, is fixed a small bracket, terminating in an upturned point, which forms a gauge of the height of the oil level. The instrument is supported on a tripod stand provided with levelling screws

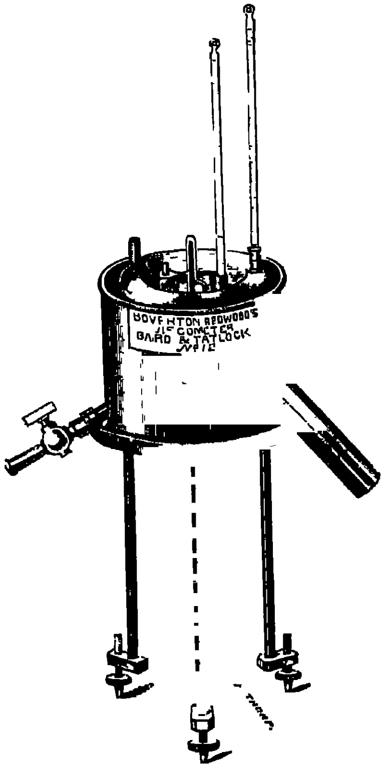


FIG. 29.—REDWOOD'S VISCOMETER.

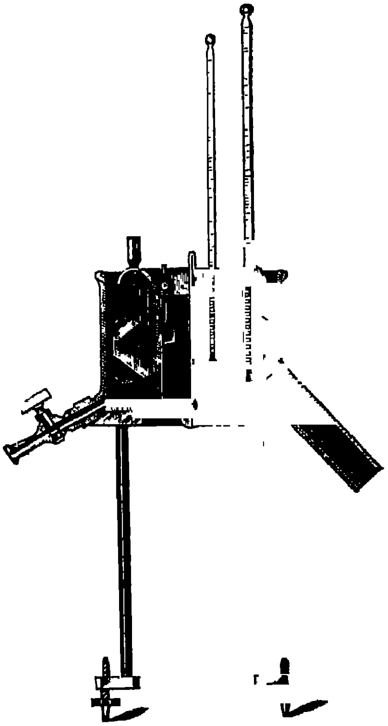


FIG. 30.—REDWOOD'S VISCOMETER.

135. *Directions for Use.*—The bath is filled with a suitable liquid to a height roughly corresponding with the point of the gauge in the oil cylinder Water answers well for temperatures up to 200° F., and for higher temperatures a heavy mineral oil may be used The liquid having been brought to the required temperature, the oil to be tested, previously brought to the same temperature, is poured into the oil cylinder, until the level of the liquid just reaches the point of the gauge A narrow-necked flask, holding 50 c c to a point marked on the neck, is placed beneath the jet in a vessel containing a liquid of the same temperature as the oil. The ball valve is then raised, a stop-watch is started at the same time, and the number of seconds occupied by the outflow of the 50 c.c is noted. It is of the greatest importance that the oil cylinder should be filled *exactly* to the point of the gauge, after inserting the thermometer, and that the given temperature should be *precisely* maintained during the experiment, a difference of ½° F. making an appreciable difference in the viscosity of some oils It is also essential that the oil should be quite free from dirt or other suspended matter, and from globules of water, as the jet may otherwise be partially obstructed. If the oil cylinder requires to be wiped out, paper rather than cloth should be used, as filaments of the cloth may be left adhering When oils are being tested at temperatures much above that of the laboratory, a gas flame is applied to the copper heating tube, and the agitator kept in gentle motion throughout the experiment The jet should be carefully examined before the apparatus is used, and, if necessary, a piece of soft string should be passed through it. The apparatus should be adjusted by means of the levelling screws, so that a spirit level, placed on the top of the oil cup, shows it to be horizontal.

136. Method of Expressing Viscosity Results.—Mr Redwood recommends that the number of seconds occupied in the outflow of 50 c.c. of the oil under examination should be multiplied by 100 and divided by 535 (the number of seconds occupied in the outflow of 50 c c. of average refined rape oil at 60° F). The figure thus obtained should be then multiplied by the specific gravity of the oil under examination at the temperature of the experiment, and divided by 915 (the specific gravity of refined rape oil at 60° F); the necessary correction for specific gravity being thus made, the final figures will express the viscosity of the oil, at the temperature of the test, in terms of viscosity of refined rape oil at 60° F.

For purposes of commercial testing, the specific gravity part of the calculation is often neglected, and where a number of samples are to be tested, the same volume of each is run through the viscometer at the same temperature, and the number of seconds occupied by this operation is taken as the measure of relative viscosity.

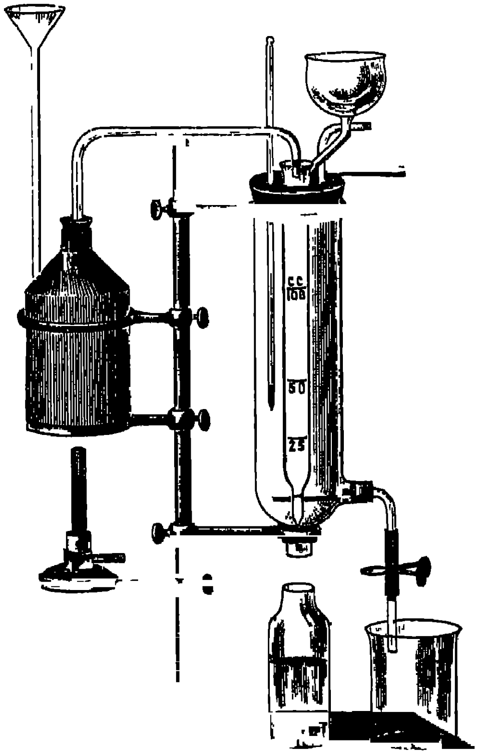


Fig 82.—The Coleman-Archbutt Viscometer.

137. The Coleman-Archbutt Viscometer—Another very convenient and rather less expensive viscometer is the Coleman-Archbutt instrument, which is here illustrated as supplied by Messrs. Baird and Tatlock, at the price of £3 3s.

This instrument consists of a glass tube, contained in an outer jacket, with a funnel for pouring in hot or cold water, a tube for running off the water, and a stirrer for mixing the water contained in the jacket In this instrument the oil used in the experiment is measured in the burette tube, and the use of a graduated vessel in which to measure the oil—after delivery—is dispensed with. As the tube containing the oil is graduated, a test can be made with either 100 c.c., 50 c c, or 25 c c If the viscosity is high, 25 c c may be sufficient for the test, if a medium oil is under examination, 50 c.c. is used, while if the viscosity is low, the full 100 c c are employed. In this apparatus samples of oil can be tested at temperatures up to that of boiling water, which is sufficiently high for most commercial purposes

The method of using the Coleman-Archbutt Viscometer is described as follows in Archbutt and Deeley's work on "Lubricants and Lubrication": The tube and jet are first carefully cleansed and dried; the jet is then closed by means of a small peg of soft wood Water at the proper temperature is next poured into the jacket, and the oil, previously strained, if necessary, and brought up to the same temperature, is poured into the oil tube up to about half an inch above the mark from which it is intended to run The oil is stirred with a thermometer, avoiding the formation of air bubbles, until the temperature is exactly correct, the final adjustment being easily made by slightly raising or lowering the temperature of the water bath. The thermometer is then taken out of the oil, which is allowed to rest until perfectly still. The wood peg is withdrawn, and the time taken by the oil to flow down to the zero mark is measured by a stop-watch, which is started as the surface of the oil passes the upper mark. During the experiment, the temperature is maintained constant by pouring hot or cold water into the jacket through the funnel, and running off the excess through the side tube as often as required, using the stirrer frequently The temperature of the water in the outer jacket is indicated by a thermometer, which remains suspended, as shown, during the whole of the experiment The open end of the oil tube should be covered by a small inverted beaker, so as to prevent water being splashed into the oil.

In making an experiment at 212° F., the cold water in the jacket is first raised gradually to about 180° F., by pouring in hot water through the funnel Then the funnel is replaced by the steam tube connected to the steam generator shown at the side, and steam is blown into the water jacket. When the water boils, the level of the water is reduced slightly to prevent splashing, and the water is kept gently boiling throughout the test When operating at this temperature, the oil is poured into the oil tube just before the water begins to boil, and the tube covered by the inverted beaker

137a. Specific Gravity of Oils.—The specific gravity of oils is best ascertained by means of the Westphal specific gravity balance (see paragraph 18)

138. Comparison of Samples of Lubricating Oils.—For lubricating purposes, the chief consideration is the viscosity of the oil It is possible to alter the specific gravity of an oil by adulteration or admixture, but where the oil is submitted to the viscosity test, the specific gravity, for commercial purposes, may safely be regarded as a secondary consideration. Where two samples of oil are tested for viscosity only, it is safe to assume that the sample which shows the highest viscosity is the most valuable as a lubricant. There is one qualification to this statement, and that is with regard to the flash point. The flash point of the highest viscosity oil should be higher than that of the oil showing a lower viscosity In a nutshell, the considerations are as follows: If two oils show the same flash point, and A has a greater viscosity than B, then A is the best oil

139. Oils for Paint Making.—It is scarcely possible, in the space here available, to enter minutely into the subject of the complete analysis of oils for use in paint making, nor would it be quite in place in a work of this character. If more detailed information is required, reference should be made to the standard works on the subject, the best of which is Mr. George H Hurst's "Painters' Colours, Oils, and Varnishes," published by Griffin and Company, London. For the purposes of this manual, a terse summary of the methods generally employed in the commercial testing of the commonly used oils, and the detection of adulteration, must suffice

LINSEED OIL

The principal tests applied to linseed oil (the oil which is of general use in paint making, are—(1) specific gravity; (2) flash point; (3) detection of adulteration by admixture of mineral or rosin oils

140. Specific Gravity —This is best ascertained by the use of the Westphal specific gravity balance, a description of which, with instructions for use, will be found in paragraph 18. The oil should be cooled, if necessary, to 60° F, and several tests should be made on the same sample

The specific gravity of raw linseed oil should be about 0·932; if under 0·930, adulteration with mineral or other oils is to be suspected If the figure obtained is over 0·937, adulteration with rosin oil is indicated In the case of boiled linseed oil, the specific gravity figure should be about 0·945, a higher figure indicates probable adulteration with rosin oil, while a lower gravity than 0·940 shows that mineral and fatty oils may have been used as adulterants

141 Flash Point.—Linseed oil flashes at about 470° F, this figure applies both to raw and boiled oil For the estimation of the flash point the Abel-Pensky apparatus should be used If adulteration with rosin oil has taken place, its presence is indicated by the fact that it flashes at a lower temperature than linseed oil (from 300° F. to 330° F.), and this constitutes a good test for detecting the adulteration. Further, if rosin oil is present, its odour will be detected during the testing operation. If mineral oils have been added, these will flash at 380° F. to 420° F. The order of flashing would therefore be as follows.—

Rosin oil, if present .	about 300° F
Mineral oils, if present .	about 380° to 420° F
Linseed oil, if present	about 470° F.

142. Adulteration with Mineral Oils, &c.—To detect the presence of rosin or mineral oil in linseed oil, the following simple test is given by Hurst: Place a little of the raw or boiled linseed oil in a beaker, add a little of a solution of caustic potash in alcohol, and boil for some time. Pour the solution into water; if the oil be pure, a clear mixture will result, if it contains mineral or rosin oil, the mixture will be cloudy or turbid;

TURPENTINE.

143 Specific Gravity—For commercial purposes, the principal test is that of specific gravity The average gravity of turpentine is about 0 867 Adulteration with petroleum spirit, rosin spirit, or shale naphtha will cause this figure to be departed from, and this deviation is a very reliable indication of adulteration It is not here necessary to describe methods for the detection of the precise nature of the adulterant, a close watch should be kept on the reading of the specific gravity balance, which, for commercial purposes, is the simplest method of testing.

144. Flash Point.—The flash point of unadulterated turpentine is from 90° to 100° F, and this figure should not vary much in pure samples. The flash point is ascertained as described in paragraph 128.

CHAPTER VI.

Gas Residuals and Bye-Products.

SPENT OXIDE.

145. The spent oxide of the gasworks is utilised for the manufacture of vitriol, and, latterly, it has been used as a source of Prussian blue The sulphur contents, however, are the most valuable constituent, and, as the material passes through the hands of agents and brokers before it reaches the vitriol maker, and further, as the moisture and sulphur contents are subject to very great variation, it is necessary that their estimation should be carefully and accurately carried out

As already mentioned, spent oxide is a very variable material. It is handled in every degree of moisture, from a very dry oxide, containing 10 to 15 per cent. of water, to an oxide which actually may contain some 60 to 70 per cent The sulphur contents are equally variable, and while one sample may test 60 per cent. of sulphur, another may be as low as 20 per cent. In colour, too, the difference in samples is very noticeable, ranging from a light brown to a very pronounced blue—in the latter case the oxide will probably contain a fair percentage of Prussian blue.

146. Sampling.—The method of sampling described in the testing of coal may be employed in the case of spent oxide also. A large sample should be taken from the bulk, and this reduced, as described, by quartering, the ultimate working sample being about a pound of the material. This is ready for laboratory use

Empty the sample out on a sheet of stout brown paper. If it is a fairly dry sample, there will be no difficulty in handling it, and the following is the best course to pursue in order to obtain the best results: Take a large porcelain mortar, clean, and dry Select all the large, hard lumps of oxide, and crush them roughly in the mortar—do not grind up, but simply crush Reduce all the lumps in this way, and then mix thoroughly Now make a heap, with the top flattened, as in the case of the coal sampling above referred to, and again reduce by quartering, to a working sample of about 200 grammes This should be kept in a sample bottle, tightly stoppered.

147. Estimation of Moisture.—Weigh out 10 grammes accurately Spread out the oxide in a dish, or on a clock glass, and dry at 100° in the water oven, weighing at intervals until constant. The loss on drying, when multiplied by 10, gives the percentage of moisture contained in the sample.

148. Sulphur.—The sulphur contained in the spent oxide is estimated by dissolving out by means of carbon bisulphide and weighing the sulphur thus obtained This operation is performed on the dried sample, and the result calculated on the sample as received Sulphur in spent oxide was formerly estimated by exhausting the oxide in an extraction tube, using cold bisulphide, and allowing the bisulphide to run right through the oxide packed in the tube, using fresh solvent for each washing By this method an excessively large quantity of solvent was necessary in order to extract the whole of the sulphur contained in the sample, and this involved a correspondingly tedious evaporation of the solvent in order to weigh the sulphur obtained

149. Two separate forms of apparatus are here illustrated for the estimation of sulphur in spent oxide. In both cases the principle is the same Fig. 34 shows a Soxhlet extraction apparatus on the left, consisting of a small flask, a Soxhlet's fat extractor, and a vertical condenser. This apparatus is supplied by Messrs. Townson and Mercer.

150. The form of sulphur extraction apparatus, illustrated in Fig. 33, known as Bale's apparatus, is also supplied by Messrs Townson and Mercer, price 17s. 6d This is a very convenient arrangement, and is used as follows · Take the whole of the dried 10 gm (paragraph 147), and rub up in a mortar. Do not grind to a very fine powder, but reduce any hard lumps, and aim at getting the bulk of the sample into pieces not larger than a pin head. A certain amount of fine powder will, of course, be included, but if the precaution of not grinding to fine powder is observed, the extraction will proceed much more satisfactorily. Weigh off one-third of the dried and crushed sample. Now dry and weigh the flask, without the tube, and note the weight of the flask alone The tube should also have been dried in the water oven at the same time as the flask, and a little cotton wool should also have been dried Now take some of the wool, and plug up the bottom of the extraction tube. This tube fits into the neck of the flask, the tube and the flask neck being ground The cotton-wool plug must fit easily, but not very tightly, or trouble will result later Having introduced the wool plug, take a slip of paper, and double it Place the weighed sample in the

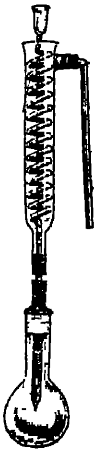


FIG. 83.—BALL'S APPARATUS

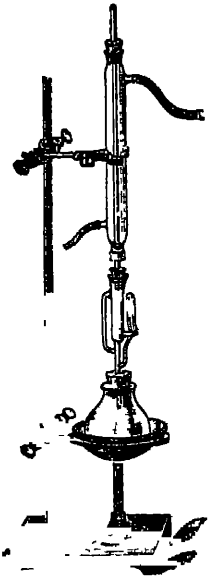


FIG. 84 —SOXHLET EXTRACTION APPARATUS

folded paper, introduce the end into the mouth of the tube, and allow the oxide to run down, taking care that none of it finds its way out through the vapour outlet at the side, near the ground top of the tube The whole of the oxide having been packed in, give the tube two or three light taps on the bench, and introduce a second wool plug on top of the mass. This second plug must also be lightly packed; if it should be at all tight, it will interfere with the working of the apparatus. Its purpose is merely to prevent the oxide from being washed out through the vapour outlet into the bisulphide flask.

The flask having been accurately weighed, and the extraction tube properly packed as described, the extraction may now be proceeded with. Place the tube into the flask, fitting tightly in place. Take a small, dry funnel, and place in the mouth of the flask. Pour about 10 or 15 c.c. of bisulphide through the tube, allowing it to sink through the layer of oxide, when the solvent will fall into the flask, coloured a dark brown. When the bisulphide has run through, connect the apparatus together as shown, the condenser having previously been fitted with a water supply. A beaker half filled with hot water contains the flask, and a small Bunsen burner supplies the necessary heat. The water used in the beaker should be previously brought to boiling, and then just sufficient poured into the beaker to cover this to the neck, when immersed. The best arrangement of apparatus for conducting this test is to clamp the vertical condenser to a heavy retort stand by means of a clamp at the *top* of the rod of the retort stand. A ring, with wire gauze, is fitted at the *bottom* to receive the water bath, and in the centre another smaller clamp is fixed to secure the neck of the bisulphide flask. By means of the upper clamp the condenser can be raised or lowered, so that the exact height of the bisulphide flask may be adjusted, and this height should be such that a small Bunsen burner can be easily slipped underneath the bottom ring and gauze. A safety burner should be used with this apparatus, not a rose Bunsen.

The apparatus now being ready, drop the beaker, and partly fill it with boiling water, as already described. Having seen that the water is running through the condenser, and that the oxide tube is tightly fitted into the neck of the bisulphide flask, raise the water bath so that the flask is immersed. After a few seconds the bisulphide will begin to distil, and presently the vapour will condense on the end of the condenser tube, and drip back upon the oxide. If hot water is used in the beaker at the commencement, there will be no necessity for the use of the Bunsen at this stage. Allow the oxide tube to fill up with bisulphide, and then slightly drop the beaker by means of the ring, until the condensed bisulphide falls from the condenser at just the same rate that the discoloured solvent drips from the end of the oxide tube into the flask. As the water in the beaker cools, apply the small Bunsen, using a very small flame, which is quite sufficient to keep the distillation of the solvent going on. The object to be kept in view in using this oxide apparatus is, that the condensed solvent should fall into the oxide tube at the same rate that the discoloured solvent runs from the end of the tube into the flask. To effect this, some little manipulation of the water beaker and burner are necessary, but with a little practice it may easily be effected. If the oxide tube fills up with solvent, and the solvent does not run through, it is only necessary to remove the burner, drop the water bath, and allow the flask to cool a little, when the discoloured solvent will fall in a steady stream until the tube is emptied Care must be observed in the use of gas flames while conducting a bisulphide test, and, as indicated, it is only necessary to use the Bunsen occasionally with this apparatus.

The extraction of a sample of spent oxide is effected in from twenty minutes to half an hour in Bale's apparatus The completion of the extraction is indicated by the solvent running colourless from the end of the oxide tube When this stage is reached, allow the distillation to continue for several minutes, clear solvent passing through all the time; then turn out the gas flame, remove the water bath, and allow the flask to cool Detach the flask, connect it to a condenser with receiver, and distil off the bisulphide until the sulphur is left in a dry condition at the bottom of the flask. Take the flask away, and place it in a cool place where there are no gas flames, allow the vapour to clear off, and then dry in the water oven, weighing after about an hour, and then at half-hour intervals until the weight is constant. The percentage of sulphur contained in the sample is then calculated as follows:—

Sulphur × 30 = per cent sulphur on sample as received.

In conducting the sulphur extraction test described above, a beaker has been mentioned as forming a convenient water bath. Beakers, however, are easily broken, and therefore it is well to use a copper vessel, made in the form of a beaker, as a water bath. The naked flame should *never* be used when bi-sulphide is employed in any test

If the Soxhlet extraction apparatus shown in Fig. 34 is employed, the weighed sample of oxide must be enclosed in a cartridge of filter paper, and placed in the cylinder of the Soxhlet extraction tube. Solvent is then poured over, and allowed to percolate through the sample, the extraction being carried on in much the same manner as in Bale's apparatus, but more solvent is required—sufficient to fill the tube up to the siphon, and still leave a quantity in the distillation flask.

SULPHATE OF AMMONIA

Sulphate of ammonia is generally tested for moisture and percentage of ammonia Commercial samples of sulphate should be of a good grey colour, and should contain 24 per cent. of ammonia

151. Moisture.—Mix the sample well, crushing the lumps in a mortar, and draw a sample by quartering, as already explained in paragraph 68 From the final sample weigh off 10 grammes on the balance, spread out in a weighed basin or on a clock glass, and dry in the water oven at 100° C After drying some time, weigh off, again dry for an hour, and again weigh, repeating the operation until the weight is constant. The loss on the 10 grammes, multiplied by 10, is the percentage of moisture.

152. Ammonia—Weigh off 2 grammes of the crushed and mixed sample, before drying Take a flask of about 500 c.c. capacity, and insert a rubber stopper pierced with one hole in the centre. Into this fit a piece of glass tubing, bent at right angles twice. To the other end of this bend fit an extraction tube with a wide end. Insert the 2 grammes of powdered sample into the flask, which should not be supported by a ring, but clipped by a stout clamp at the neck. Pour into the flask about 200 c.c. of tap water, and 20 c.c. of milk of lime, made by rubbing up slaked lime to a paste with water, and shaking well. Into a small beaker, holding about 200 c.c., run 50 c.c. of normal sulphuric acid solution, and arrange the apparatus so that the end of the gas delivery tube dips underneath the surface of the acid about half an inch.

When all is ready, apply a gentle heat by means of a small rose burner, and bring the contents of the flask to the boiling point. The lime will froth up during the distillation, and the flask should be of such a size that, when containing the bulk of liquid indicated, there is plenty of room for the frothing.

Care must be taken in this test that draughts are excluded from the vicinity of the apparatus during the distillation It frequently happens, when the flame of the burner is wafted aside by a current of air, that the acid in the absorbing beaker is sucked up through the tube and into the distilling flask, of course spoiling the test altogether. If the acid ascends beyond a safe point in the tube, raise the gas flame for a second The rush of vapour will drive the liquid back, and the flame may be adjusted again. The distillation should be continued at a brisk rate—provided there is no smell of escaping ammonia—for about half an hour At the end of this time, drop the beaker of acid, turn off the gas, and allow the acid to cool. Then titrate with normal soda solution, using methyl orange as an indicator. The number of c c. of soda solution used are deducted from the original volume of acid taken, and the difference is the acid neutralised by the ammonia in 2 grammes of the sulphate. The calculation is then as follows —

$$(\text{C C. acid taken for absorption} - \text{c c soda used in final titration}) \times 50 \times 0{\cdot}017 = \text{per cent. ammonia}$$

GAS LIQUOR.

The following method of estimating the ammonia may be used in the testing of the ammoniacal liquor from gasworks, and also the "crude concentrate" used in many chemical manufactures

153. Specific Gravity —This may be taken by the Twaddell hydrometer. Gas liquor is usually stated as so many degrees Twaddell, and concentrate as containing a certain percentage of ammonia The Westphal balance may be used, or the method of estimating specific gravity by actual weighing, if greater accuracy is required.

154. Percentage of Ammonia.—Mix the sample well, and pipette off 25 c.c into the apparatus used for testing sulphate of ammonia, weighing the sample bottle before and after. Distil as there described (paragraph 152), and titrate with normal soda, using methyl orange. Each c.c of acid consumed is equal to 0·017 gramme of ammonia, therefore

$$\frac{\text{c.c. acid consumed} \times 100 \times 0{\cdot}017}{\text{Weight of liquor taken}} = \text{per cent. ammonia.}$$

155. Ammonia in Ammonia Concentrate.—Prepare the ammonia distillation apparatus as described for sulphate of ammonia Clear the neck of the sample bottle containing the concentrate to be tested, removing all sealing wax, etc. Weigh on the rough balance Now place about 150 c c. water in the distillation flask, and pipette off 25 c c. of the concentrate into the flask, allowing the pipette to discharge under the water. After withdrawing the pipette from the sample bottle, the latter must be carefully stoppered again Weigh the bottle, and so ascertain the weight of concentrate taken for the test. Now conduct the distillation as before, with milk of lime, titrating with normal soda solution

156. Ounce Strength.—A common method of stating the strength of crude gas liquor is by what is known as "ounce strength" This term indicates the number of ounces by weight of real sulphuric acid, which would be neutralised by a gallon of the gas liquor. The most accurate method of estimation and expression, from the standpoint of the analyst, is to indicate the percentage of ammonia contained in the liquor (see paragraphs 152-5), but as the "ounce strength" is very often used in practice, the method of testing is here described.

Solutions Required—Sulphuric acid (55 1 c c. pure acid of exactly 1 84 specific gravity, per litre) Measure off 55·1 c c. of the strong acid, and pour it, with constant agitation, into water in a litre flask, making up to the mark when cool. Test this solution with the Westphal specific gravity balance (paragraph 18) at 60° F.; it should indicate 1·0644 s.g Such an acid is known as "10 per cent acid."

Alkali Solution.—A solution of ammonia is generally used (117 c.c. liquor ammonia 0 880 specific gravity, in a litre). 117 c.c of pure liquor ammonia is diluted to a litre Pipette off 10 c.c. of this solution into a conical beaker, add a little water and a few drops of methyl orange, and titrate with the "10 per cent acid." The two solutions should exactly neutralise each other

Method of Ascertaining Ounce Strength.—Fill a burette with the gas liquor, and run off 16 c.c. into the flask used in paragraph 152; add milk of lime, and distil as there described. Absorb the ammonia in 16 c.c. of "10 per cent. acid," diluted with water to a convenient depth. Continue the distillation until the contents of the flask are free from ammonia Cool the acid distillate, add a little methyl orange, and titrate with the ammonia solution until the excess of acid is neutralised. Deduct the number of c c from 16, and the figure so obtained is the "ounce strength"

157 Free and Fixed Ammonia in Gas Liquor.—The proportions of free and fixed ammonia are ascertained by (1) distilling the liquor without lime, and titrating as in paragraph 152 This gives the free ammonia, the distillate being titrated separately (2) Now add to the residue in the flask, after boiling off the free ammonia, the milk of lime, and continue the distillation, absorbing the gases in fresh acid. Titrate this separately also; the result is the fixed ammonia.

158. Ammonia by Direct Titration—Pipette off 25 c c of liquor into a medium-sized flask, dilute well with water, so as to diminish the colour of the liquor. Add methyl orange, and titrate with standard acid Ensure neutralisation by using litmus paper at the end of the titration, and make several tests, taking the average of two or three Then,

c.c standard acid used × 4 × 0 017 = per cent. of ammonia in liquor.

159. Prussian Blue in Spent Oxide.—Weigh off 2 gm. of dried sample in a beaker, add 50 c c warm water, and digest on the water bath for an hour. Filter, wash the oxide on to the filter paper, and wash well with hot water This preliminary digesting and washing removes the sulphocyanides Now wash the oxide back into the beaker, cover with caustic soda solution of 2° Tw strength, and digest on the water bath for about two hours. Filter through the paper already used, transfer the oxide to the funnel, and wash well with hot water Dilute the filtrate and washings a little, and add a mixture of ferrous and ferric chloride, and acidify with hydrochloric acid The Prussian blue is now precipitated. Transfer the whole to a very large beaker, and dilute largely Allow to stand over-night Next morning, siphon off the clear liquid as far as possible with care, but avoid agitating the precipitated blue Take a clean weighing bottle, and weigh it. Into the water bath place a filter paper, dry, and weigh in the weighing bottle After drying and weighing, fit the paper into a funnel, and filter off the blue. Dry the filter paper and blue (still in the funnel in the water oven), and weigh in the weighing bottle, which should be kept in the oven during the final drying. Cool under the desiccator, and weigh The difference in weight is the Prussian blue from 2 gm. This, multiplied by 50, gives the percentage contained in the dried sample.

COKE.

160. Ash.—Weigh off 1 gm. of the finely-powdered sample, and estimate the ash as in paragraph 71. The residue, multiplied by 100, gives the percentage of ash.

161. Sulphur.—This is estimated as in paragraph 72.

162. Moisture.—The sample should be roughly powdered, and 10 gm dried in the water bath until the weight is constant. The loss, multiplied by 10, gives the percentage of moisture in the coke.

163. Calorific Value.—This is estimated as in the case of coal (paragraph 74).

TAR.

164. When coal tar is to be tested, the examination usually comprises the estimation of the following constituents.—

(1.) Ammoniacal liquor
(2) Light oils.
(3) Middle oils
(4.) Anthracene oils.
(5) Pitch.

The tests should be conducted on as large a scale as possible, and where many tests are required a small still is generally fitted up specially For an occasional test, however, a distillation flask of about 2 litres capacity, provided with a side neck, will answer. Fit up the flask attached to a Liebig's condenser, so arranged that warm water can be used in the condenser jacket in place of cold, at the end of the test. Dry the flask, and weigh it. Measure about 2 litres of the tar into the distillation flask, and weigh the flask and tar; the difference gives the weight of tar taken for the test.

Fit a rubber stopper, bored with one hole, into the flask. Through the hole introduce a nitrogen-filled thermometer, with a scale up to 400° C., with the bulb on a level with the side delivery tube. Support the flask on a piece of wire gauze, and clamp the neck of the flask firmly. Connect up the side tube of the flask to the condenser, place a flask, immersed in cold water, to receive the distillate, and commence heating with a good rose Bunsen burner

165 Ammoniacal Liquor and Light Oils —When the first drop comes over, note the temperature Raise the heat very gradually to 170° C., collecting the distillate in a receiver. The products distilling off up to 170° C. consist of ammoniacal liquor and light oils Place the mixture in a graduated cylinder, allow to settle, and read off the volume of each, or transfer the mixture to a separating funnel, run off the water and oil, and measure them separately The liquor is tested as previously described, and the specific gravity of the light oils is estimated by the Westphal balance

166. Middle Oils.—After the distillation of the first fraction, warm water must be run into the condenser in place of cold, and the temperature raised to 270° C After 170° C. is passed, the products of the distillation generally have a tendency to block up the delivery tube from the flask, in this case the tube must be gently warmed by the application of a Bunsen burner, and the water in the condenser kept hot. Afterwards the distillate becomes more fluid, of a yellow colour, and of a pasty consistency on cooling During this part of the test, the receiver need not be cooled. When the distillation ceases keep the distillate liquid by immersion of the vessel in hot water, and take the specific gravity while hot with the Westphal balance, noting the temperature at which this is done While liquid, measure off the distillate.

167. Anthracene.—Take away the condenser, and in its place use a short, wide tube, and continue the distillation to about 360° C, when dense fumes are given off If any of the last distillate solidifies in the tube, heat this, and allow the liquid to run into the receiver. Keep the distillate liquid by standing the receiver in very hot water, and measure while liquid. Take the specific gravity at the same time (at about 75° C)

168 Pitch.—Allow the flask to cool, and then weigh; deduct the weight of flask, and the difference is the weight of the pitch, which should be melted out and kept for reference.

From the figures obtained in these tests, the approximate yields of the different products may be calculated.

CHAPTER VII.

Chemicals and Raw Materials Used in Paper Making.

Although the paper maker has made considerable progress during recent years, so far as the testing of his raw materials is concerned, it is nevertheless true that in many works little is done in the way of controlling the various raw materials employed in the manufacture, the chemicals which are used, or the fuel and oil consumed in such great quantity.

It is quite true that in some paper mills everything is done which can be desired to ensure proper control in every direction, not merely so far as the raw materials are concerned, but this control is extended to the process operations generally, and every step of manufacture is watched by a competent chemist There are paper mill laboratories in Lancashire, in the heart of the paper-making district, which can only be described as models, and the equipment of which leaves nothing to be desired, either as regards apparatus or staff. But it is equally true that other mills, not far removed as regards locality, are so badly equipped that chemical knowledge is as conspicuous by its absence as is the chemical balance; where the smallest weight to be found in the place is ¼oz.; where, if bales of pulp are to be weighed for testing purposes, the analyst is referred to the weigh-bridge in front of the lodge; and where the most delicate balance resolves itself into what appears to be a pair of old grocer's scales

In many mills in the North, the necessity for establishing chemical control of raw materials has only been realised, in any general degree, within a very recent period. Even five years ago there were many large and very important mills where no testing whatever was carried out. The condition of the paper mills in the same district to-day is very different There are, as already remarked, several thoroughly equipped laboratories, and many others of more modern installation and recent development, but, when so much has been said, it yet remains a fact that, in a large proportion of mills, the chemical control is very imperfect, if it exists at all

The introduction of wood pulp is responsible, in a number of cases, for the awakening of the paper maker, so far as chemical analysis is concerned. It was indeed a rude awakening for him to find that he was paying for *water* at the rate of some £10 a ton, and, once this fact had gone home, and it was realised that pulp makers were in the habit of delivering dry pulp containing as much as 15 per cent of moisture (and in many cases even more) it was not long before efficient steps were taken to alter this state of things, and to ensure that the paper maker only paid for the exact weight of fibre which he actually received. At the same time, it is astounding, to those who are intimately acquainted with the circumstances, to observe the very gradual manner in which the paper maker has availed himself of chemical assistance in controlling the condition of what is now one of his staple raw materials He has manifested a most sublime faith in the quality of the goods delivered to him, and he has only recently appreciated the fact that, by intelligent testing, it is quite possible to effect economies, the influence of which, at the end of the year, is little short of startling, and the instances mentioned in Chapter I. might be multiplied indefinitely

The testing of pulp, and the results obtained, paved the way in many mills for the introduction of other tests. In some instances, however, the initiative in the development of the analytical department of the paper mill has *not* come from the management There are many cases where, on the part of a member of the office staff, the possession of a certain amount of chemical knowledge, acquired laboriously through the medium of the technical school, and in the hands of one acquainted with the practical requirements of a paper mill, has resulted in very beneficial effects on the economy of the establishment.

This latter point is one which will bear emphasis. Paper makers, when told that they should avail themselves of chemical knowledge, often reply that it is not within their reach; public analysts' fees prohibit their services being regularly employed, and they cannot afford to retain a chemist as a permanent member of their staff The difficulty has been solved in several mills: the manufacturer has found that one of his clerical assistants has chemical tastes, a sufficient amount of analytical knowledge to carry out simple tests, and—what is equally valuable—an acquaintance with the *practical* conditions of the mill which has enabled him to direct his energies into the right channels Such a man has been given a free hand to apply his knowledge, and the result has been eminently satisfactory. The raw materials have been tested—pulp, bleach, and other chemicals—and when the quality has been at fault, a public analyst has been called in to confirm the works test, and to fight the claim Other paper makers might adopt this course; the result could scarcely fail to be satisfactory The cost of fitting up a small laboratory is very little; the benfits to be derived from simple, intelligent work, are very great.

With these introductory remarks, the testing of some of the raw materials and chemicals used in the paper mills may be considered, with the note that in the other sections will be found many tests equally applicable to the paper trade.

CHINA CLAY.

169. Sampling.—Draw a representative sample from the bulk by taking a quantity from a number of the packages—every third one, for instance—thoroughly crushing, mixing, and sampling, as in the case of bleaching powder (paragraph 85)

170. Moisture.—Weigh off 50 gm of the prepared sample, and dry in a large dish in the water oven at 100° C., weighing at intervals of about an hour till the weight is constant. The loss, multiplied by 2, gives the percentage of moisture.

171. Colour, etc.—The physical properties of China clay are judged by comparison with good standard samples.

CAUSTIC SODA.

172. Sampling.—See paragraph 93.

172a. Available Alkali.—See paragraph 94.

172b. Insoluble.—See paragraph 95.

SODA ASH.

173. Sampling.—See paragraph 104.

173a. Insoluble Matter.—See paragraph 105.

173b. Available Alkali.—See paragraph 106

RECOVERED SODA.

174. Available Alkali.—Recovered soda must be very finely powdered, and the sample boiled in water before the titration for total alkali is made, as in the case of soda ash.

GELATINE

175. Moisture.—Weigh off about 50 gm. of the sample (obtained by taking samples from various parts of the delivery), break into small pieces, and dry in the water oven at 100° C. until constant. The loss, multiplied by 2, gives the percentage of the moisture present

176. Ash.—Weigh off 10 gm. of the carefully selected sample, and burn off in a weighed platinum dish; ignite the residue at a dull red heat over a Bunsen burner or in the muffle furnace until combustion is complete, and residue × 10 = per cent. ash.

177 Strength.—A method for estimating the relative strengths of samples of gelatine is to make up jellies with water, using the same weight of sample and the same volume of water in each case When the jellies are cold, they are tested by placing weights upon the surface By the behaviour under these conditions comparative values may be obtained for the "stiffness"

STARCHES

Apart from the microscopical examination of starches (which does not come within the scope of this volume), the chemical tests usually applied are those for moisture and ash.

178. Moisture.—Draw a fair sample (see gelatine); grind and mix thoroughly, and ignite 10 gm in a platinum or porcelain basin in the water oven at 100° C, until the weight is constant. The loss, multiplied by 10, gives the percentage of moisture.

179. Ash.—Weigh off 10 gm in a platinum crucible, and ignite over a Bunsen burner or in the muffle furnace until combustion is complete, and the weight is constant; cool in the desiccator, and weigh The loss, multiplied by 10, gives the percentage of ash.

Note—In the estimation of ash in organic substances, such as starch and gelatine, the ignition must be continued until *all* the organic matter is destroyed, and only the mineral residue remains. Where the percentage of ash is small, as in starch [illegible] (about ½ per cent) errors may easily be introduced by not ensuring complete combustion

180. Strength.—Estimate as in the case of gelatine (paragraph 177).

181. Insoluble Matter.—Dry and weigh a filter paper Weigh off 10 gm. of the prepared sample, and dissolve completely in water. Filter through the weighed paper, well wash the residue with hot water on the paper, and dry the paper (still in the funnel) in the water oven. Weigh off in the weighing bottle. Multiply the residue by 10, this gives the percentage of insoluble matter present If this is small, a check test should be made on 50 or 100 gm.

ALUM SULPHATE OF ALUMINA ALUM CAKE

182. Alumina.—Weigh off 2 gm. of the powdered and thoroughly-mixed sample, dissolve in water in a beaker, add ammonium chloride and ammonia in excess, filter, wash the precipitate well on the filter, dry on a clock glass over the water bath, finish in the water oven, ignite until constant, and weigh the ignited residue This × 50 = per cent alumina

183. Insoluble Matter.—Weigh off 100 gm, and dissolve in hot water. Filter, wash the residue, and dry the filter paper over the water bath or in the oven, and ignite in a weighed crucible The residue is the percentage of insoluble matter

184. Free Acid.—The method generally employed for the estimation of free acid is to digest, say, 10 gm of the finely-powdered sample in alcohol, filtering, and titrating the acid with standard alkali

A solution of Congo-red is turned blue with free acid This is useful as a qualitative test

PEARL HARDENING.

185 Moisture—Grind, and well mix the sample Weigh off 10 gm., and heat in the water bath to 260° C., until constant The loss, multiplied by 10, gives the percentage of total moisture

186. Colour, etc—The physical properties of pearl hardening must be estimated by comparison with good standard samples

BLEACHING POWDER

186a.—See paragraphs 84, 85, and 86.

BLEACHING LIQUOR.

186b.—See paragraph 87

PAPER TESTING.

187 Ash.—Weigh off 1 gm. of the sample into a porcelain or platinum crucible, and ignite over a Bunsen burner until the organic matter is completely burnt off. Cool under the desiccator, and weigh; ignite again, cool, and weigh; repeat this if any loss has taken place. The residue (multiplied by 100 if 1 gm has been taken), gives the percentage of ash

188 Moisture.—Where the estimation of this is required, cut up from 20 to 100 gm, dry in the water oven, and weigh until constant Calculate the moisture from the loss.

189 Physical Tests—Physical tests are somewhat outside the scope of this work For information on the subject the reader is referred to "A Text-book of Paper Making," by Cross and Bevan (E. & F N. Spon).

Thickness.—This is best estimated by means of a micrometer, the thickness being read off direct from the scale of the instrument

Weight—Special balances are in use in most paper mills, and need not be described in detail.

Strength—The remark in the last paragraph also applies here

190 Detection of Mechanical Wood Pulp.—A solution of phloroglucin in hydrochloric acid gives a red colour when mechanical pulp is present The percentage of this constitutent is estimated in some mills by comparison with a set of samples containing known percentages of mechanical pulp.

THE SAMPLING AND TESTING OF WOOD PULP.

191 Perhaps no raw material used in any process of manufacture is liable to greater variations in physical condition than wood pulp By reason of its composition, pulp is subject to variation in a measure which is almost bewildering, so far as its moisture contents are concerned As is well known, the pulp is manufactured in the wet condition, and is handled commercially as either wet pulp or dry pulp. Wet pulp is supposed to contain 55 per cent of moisture, and 45 per cent of absolutely dry or bone-dry pulp; dry pulp is supposed to contain 90 per cent. of bone-dry pulp, and 10 per cent of water. Besides these two divisions, pulp is known as chemical pulp and mechanical pulp. In the one case the material is obtained by the chemical treatment of the particular wood from which the pulp is manufactured, while, in the case of mechanical pulp, the operations involved in its preparation are wholly of a mechanical nature

In commerce, then, we find the following qualities of wood pulp. Moist mechanical pulp, dry mechanical pulp, moist chemical pulp, and dry chemical pulp It is with these four qualities that we have to deal

The problem of sampling and testing wood pulp, though on the face of it apparently simple, is yet one of the most difficult which the industrial chemist has to solve. Very much has already been written on this subject. The pros and cons of the matter have, at different times and in various places, been more or less completely discussed, and what is here contemplated is to set forth, as clearly as possible, a plan of working, by following which the paper maker and his chemist will be able, with a fair degree of exactness, to deal satisfactorily with most of the situations which arise in the work of testing their supplies. The methods suggested with reference to damaged parcels are the outcome of a somewhat wide experience of this difficult part of the work of pulp testing, and they have been applied, during the past five years, with a considerable amount of success

192. Percentage of Bales to be Sampled.—The regulation percentage of bales to be taken for sampling purposes is recognised as being 2 per cent. of the total number comprised in the parcel, not less than five bales being taken for the purpose of a test. Where a very small parcel is concerned—say, 100 bales, more than five bales are desirable; a good rule is to take 10 out of such a small parcel. The reason for this is, that where very small parcels are concerned, and where only five bales are taken for a test, it is quite possible that a second test will show a very different result from the first For instance, in a parcel of 100 bales, two lots of five each were taken, and sampled separately The first showed 15 per cent. of moisture, the second gave only 10 per cent. Now, if the first five bales only had been sampled, the result would have shown a claim of 5 per cent. excess moisture. If the sample had happened to consist of the second five bales, there would have been no claim whatever In such case, therefore, it is plain that it would depend entirely upon chance as to which of the results might be obtained If, however, the whole of the 10 bales were included for the purpose of the test, the average result would be much more likely to represent the true condition of the parcel of pulp. In small lots, then, experience indicates that the more bales included in the sample, the better is the test, and in small parcels, consisting of 200 bales and less, not fewer than 10 bales should be taken in sampling Where a parcel consists of anything from 500 to 5,000 bales, the specified proportion, 2 per cent, is very satisfactory

In very large parcels of pulp, from 5,000 to 20,000, the operator is confronted with a different state of things In a parcel of 10,000 bales, in order to conform with the provisions of the existing contract note, at least 200 bales must be sampled, and, where the higher number is concerned, 400 bales will be required for the purpose of testing It may seem almost out of the question that such large parcels as this will need to be dealt with, but as a matter of fact they are by no means unknown, and the mechanical work of sampling 400 bales of moist pulp is, to say the least of it, rather staggering

When dealing with these exceptionally large parcels, it is a matter for consideration whether less than 2 per cent of the total number of bales might not be taken in sampling At present the chemist has no option, and, if his test is to be officially recognised, he must take the full number of bales as specified in the contract note Experience seems to indicate that a deviation from this rule might be allowed with perfect safety, but, under existing circumstances, the full number of bales must be included if the test is to be recognised

193. Damaged Bales.—Where a parcel of pulp is found in normal condition, the work of sampling is a very simple one In such a case it will generally be found that, by selecting the stipulated 2 per cent. of the total number of bales in the parcel from different points in the stack, any two tests will agree fairly well, provided that the pulp is moderately uniform in moisture When, however, the pulp is received in a damaged condition, this simple state of things is altogether altered Pulp may have been damaged by sea water in transit, either by reason of having been carried as deck cargo, or from other causes, or it may have been damaged by rain water, through being packed in railway wagons, which have not been properly sheeted up during heavy rain. In either of these cases the difficulty of testing is very considerably increased. To accurately sample a damaged parcel of pulp requires a great deal of discrimination on the part of the operator.

If it is known that the parcel has been damaged, the bales should be very carefully selected from all parts of the stack, and the weights accurately taken. We will assume for the sake of example that a parcel of 2,000 bales of moist mechanical pulp has been delivered in bad condition, and is wetted by sea water. It will generally be found in such a case that the bales constituting the parcel may be divided into two classes. In the first class the weights will be high, and in the second class they will be either normal, or a little over the normal weight. Assuming that the parcel is composed of 4 cwt. bales, and that 40 bales are being taken for sampling purposes, we should probably find that some 20 bales would scale anything from a few pounds over 4 cwts. to about 4 cwts. 1 qr., while the rest of the bales selected would weigh 4 cwts., or a few pounds above or below this figure.

In weighing the bales selected for sampling, the two classes above referred to would generally be found scattered throughout the parcel. Thus it might happen that the first half-dozen bales would scale normal weights, then a heavy bale or two would come in, after which normal bales, wet bales, and dry bales might be indiscriminately mixed. The point is, that when the whole 40 bales had been weighed, and the weights examined, it would be seen that the selected bales could be divided into the two classes already mentioned—normal bales and abnormal bales. The normal bales, when opened, will be found to be fairly uniform in moisture contents; the abnormal bales, on the other hand, generally prove to contain a thick layer of top and bottom sheets which have been saturated with sea water. In very bad cases the damaged pulp is not merely at the top and bottom of the bale, but the water will have penetrated quite through, and the pulp to all appearances will be uniform in moisture, but the percentage of moisture, instead of being 55 per cent., will be nearer 60, or even 70 per cent. This excess of water, however, is often found, not always evenly distributed through the bale, but in some cases, for a depth of a couple of inches the sheets will be quite saturated; in the rest of the bale a ring of pulp in the centre of the sheet is in about normal condition, while the remainder of the sheet will be saturated with water taken up during transit.

This is a most difficult state of things to deal with The shipper of the pulp in such cases as this sometimes argues that all damaged bales should be rejected in sampling, and that only bales of normal weight should be included for the purpose of the test From the seller's point of view this may seem a fair contention, but to anyone who has had an extensive experience of damaged parcels of pulp, it is quite impossible to admit the argument. If, in a parcel of 2,000 bales of moist mechanical pulp, it is found on sampling that 20 bales scale, as already indicated, up to 28 lbs excess weight—for the sake of argument we will assume that the average of excess weight in this parcel is 20 lbs per bale—then, according to the contention indicated above, *all* of these heavy bales should be rejected, but if, during a careful sampling of this parcel, out of 40 bales selected for the test, half of them show excess weight, then it is only reasonable to assume that something like this proportion of heavy bales will be found throughout the consignment

The seller's reason for taking exception to the inclusion of heavy bales in a test is that the percentage of moisture so obtained will be unduly high, while, if the heavy bales are kept out of the sample, the percentage of moisture will be nearer the normal, and accuracy will be ensured.

On the other hand, the paper maker argues that if the heavy and, therefore, wet bales are rejected in sampling, then the percentage of moisture, as indicated by such a test, will be far too low; and since it is apparent, without testing, that a considerable portion of the parcel contains a very large excess of water, it is not fair that he should be asked to pay for this pulp as containing 55 per cent. of moisture, when part of it certainly contains at least 60 or 70 per cent.

What are the relative merits of these contentions, and what is the proper course to be pursued under such circumstances? On the one hand, it is perfectly true that, if the heavy bales are included in the sample, then the moisture result will be very high, but it is equally true that the seller will obtain credit for the extra weight of the bales, and therefore the gross weight of the parcel, as calculated from the mixed bales, will be very much higher than would have been the case if the heavy bales had been rejected Therefore, although the test will show a high percentage of moisture, the gross calculated weight of the parcel will also be high, and, if the sampling is accurately performed, and, moreover, if the selection of the bales has been carried out in a perfectly fair and thorough manner, taking representative bales from all parts of the stack, it is not too much to say that the result thus obtained will give a true indication of the actual air-dry pulp contained in the parcel

Pulp merchants sometimes advance the contention that the object of testing is to ascertain the condition of a parcel of pulp *when despatched from the pulp mill*, and that, therefore, only intact bales, which scale invoiced weight within a few pounds, should be included in testing This argument is one which, in practice, cannot possibly be maintained If the pulp has been shipped in good condition, and has been stored in such a manner that it does not take up sea water or rain water during transit, the parcel will be delivered in the paper mill in practically the same condition as it left the pulp mill, and the operation of sampling and testing will present no difficulties whatever If, however, the parcel has been damaged by the absorption of sea water or rain water during transit, the pulp, as delivered, will *not* be in the condition in which it left the pulp factory The question which the chemist has to decide, then, is · What is the percentage of air-dry pulp contained in the parcel when delivered in the paper mill; *not* the percentage of air-dry pulp which this same parcel contained when it left the works of the pulp manufacturer.

If the paper maker is willing to buy pulp on the strength of a test made before the pulp was shipped, he is, of course, perfectly at liberty to do so; but in commerce it seems hardly reasonable to suppose that such a proposal could for a moment be entertained seriously. Therefore the conclusion to which we are forced is this:—

In the case of damaged pulp, the bales for the test should be selected in an extremely careful manner Where 4 cwt bales of moist mechanical pulp are concerned, a good rule is to reject all bales which scale over 4 cwts. 1 qr Bales which weigh from 4cwts 1 qr to the normal weight may safely be included, it being, of course, understood that all the bales selected shall be in perfectly sound condition so far as the packing and general appearances are concerned If a bale is clearly soaked with water, then it should be rejected, but in very many cases it will be found, where 40 bales are weighed out of a damaged parcel, that bales which are perfectly sound to all appearances, and not abnormally damaged, will scale anything from 10 to 20 lbs over the invoiced weight, *without* showing distinct appearances of having been soaked with external moisture Abnormally heavy bales should be marked when weighing, a good plan being to rip up the canvas wrapper so as to call attention during the sampling, so that special care may be given to them.

It occasionally happens, even in such a case as the one under consideration, that not only are very heavy bales present in fairly large proportion, but light bales also are encountered. These light bales may weigh anything from 3 cwts 2 qrs. to the invoiced weight—that is, 4 cwts To all appearances the bales are in perfect physical condition The wrappers may be intact, and the sheets may be perfect, but on opening they will be found to consist of pulp which certainly does not contain 55 per cent. of moisture Now, it is not reasonable to reject such bales during weighing If the bales bear any indication that they are not intact, or that sheets have been lost, it of course goes without saying that they should be rejected, but, in the absence of such indications, they ought to be included when weighed haphazard in the ordinary course

The rules to be observed in sampling are then as follows.—

Normal Parcels—

(1) The bales to be selected from every part of the stack.

(2) The top and outside rows of the stack not to be sampled

(3) All bales to be sound and intact as regards wrappering and bands.

(4) Bales which are clearly of different size or shape from the normal to be rejected altogether

Damaged Parcels.—

(1.) Bales to be selected from every part of the stack
(2.) The top and outside rows of the stack not to be sampled.
(3.) All bales to be sound and intact as regards wrappering and bands.
(4.) With these provisions the bales to be selected haphazard
(5.) Bales weighing up to 4 cwts 1 qr in the case of wet pulp, and 2 cwts 1 qr in the case of dry pulp, to be included in the sample
(6.) Light bales, *if sound and intact,* to be included as encountered.

194. Selection of Bales—Assuming that the parcel to be sampled is not in any way damaged, then in selecting the bales for sampling, only sound bales in thoroughly good condition should be admitted The requisite number should be taken as nearly as possible from all parts of the stack, top bales should be rejected, as, if the weather has been dry, they may be unduly dried in, while, if any rain water has penetrated through the roof, the top bales of the stack will almost invariably have absorbed this moisture. Rejecting rejected top bales, and in all cases going as far into the stack as practicable, the bales should be made to represent, as nearly as possible, the whole of the stack.

195 Weighing Bales. — Having secured the correct number of bales, these are weighed separately For purposes of pulp testing, an accurate platform weighing machine is indispensable This is not always forthcoming, but it is certainly to the interest of the paper maker to ensure that pulp testing is performed with the aid of a thoroughly reliable machine, which will weigh up to, say, 10 cwts. This machine should be of the portable type, which can easily be removed from one part of the warehouse to another, so as to avoid excessive labour in trucking the bales. Before commencing weighing operations the machine should be accurately balanced The bales are then carefully weighed separately, weighing to the nearest pound. During the weighing operations a close watch should be kept on the brand marked on the bales, in order to ensure that wrong bales are not included in the parcel.

196. Method of Sampling.—On this point, as on others connected with the subject, many methods of procedure have been suggested It is not here intended to discuss the merits of the various processes, but to indicate a system which will, in careful hands, yield accurate results. The method described is now in use in many of the principal paper mills in the North of England, it has been adopted by the leading public analysts who make a speciality of pulp testing, and it is recognised as a standard method by both buyers and sellers of pulp The system is as follows —

Rip open the canvas covering of the bale, turn back about 2 inches of pulp, and draw out a sheet; take a second sheet from the centre, and a third from the other side, also about 2 inches from the bottom. (In large bales it is a good plan to draw *four* sheets at regular distances through the thickness of the bale; this is not officially recognised, however.)

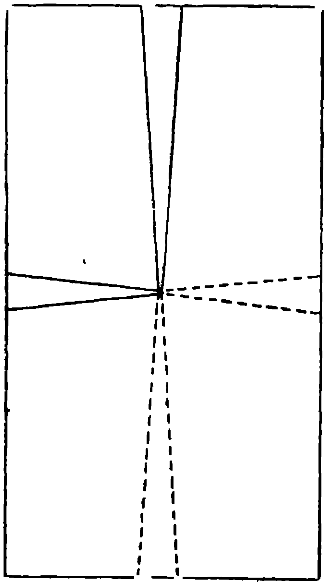


Fig 35

Having drawn out the sheets, cut from each two triangles, as shown in Fig. 34, the points of the triangle being in the centre of the sheet, and the base to measure about an inch in width. The triangles should be cut from alternate ends of the sheets.

In the first sheet, the top and left-hand side may be taken, in the second sheet, the bottom and the right-hand side are selected. If four sheets were taken, instead of three, this would make a very accurate distribution of the sampling, as all parts of the bale would be equally represented

In cutting the samples by this method, the triangles may either be cut while the sheets are in position in the bale, or sheets may be withdrawn until a number of bales have been sampled, and the whole of the triangles cut at once In this way the actual sampling is hastened, and there is less handling of the pieces.

The sampling operations must be rapidly performed, as, while the sheets of pulp are being exposed to the air, the moisture is in some degree being abstracted, according to the condition of the atmosphere For cutting samples of pulp a keen, heavy pocket knife is the best instrument to use Special sampling knives have been devised, and many have been experimented with, but so far the author has found that a heavy pocket knife, with a keen edge, answers better than anything else

The triangles, drawn as above, the one cut from the centre to the end, and the other from the centre to the side of the sheet, should be secured as nearly as possible the same size—that is, when, say twenty sheets have been sampled, the long strips, when compared, should be as nearly as possible the same width at the base of the triangle, and should taper off uniformly to the inside of the sheet It will be clearly seen that, if in cutting the first triangle, the sample is cut with a base half an inch wide, and in the second sample this base is enlarged to three-quarters of an inch, then a much greater proportion of the outside of the sheet will be included in the second sample than in the first. If this outer edge be wet, then the moisture will be greater than in the case of the first strip, while, if it be dry, a greater proportion of dry pulp will be present in the sample.

The author has seen many cases where some samples have been cut at least twice as large as others, and when, as often happens, the large samples are the wettest, it follows, of course, that the result thus obtained would be far too high in moisture, while, on the other hand, when the large samples are the driest, the moisture results will be too low. It is of the utmost importance, therefore, that these triangular samples should be cut as nearly as possible the same size The base should measure the same in each case, and the samples should taper uniformly from the base to the point Unless this precaution is observed, the result will be inaccurate

The second point is that many operators, when using this method, draw the samples all from the same end of the bale. If one end of the bale be wetter than the other, a corresponding error will be introduced, while if one end of the bale be dryer than the other, the moisture result will be too low. The only way to overcome this difficulty is to cut the samples from alternate ends of the sheets If four sheets be taken out of each bale, then the first sample should be drawn, say, from the top and left-hand side of the sheet The samples cut from the second sheet will be from the right-hand side and the bottom The third sample will be cut again from the top and left hand, while the fourth will again be cut from the right-hand side and the bottom. In this way a perfectly representative sample is obtained, and, in the present condition of things, it is not easy to suggest a more practical system

A method which has found much favour in the North of England, and the practice of which has been conducted with extremely satisfactory and uniform results, is that of taking one sample out of each sheet, this being a triangle taken at a point one-third down the sheet, and one-third across. It is no exaggeration to say that during the last five or six years many thousands of tests have been made in different kinds of pulp, where this method has been employed, and the result has, generally speaking, been very satisfactory This method is based upon the assumption that there is a point in the sheet of pulp which, under normal conditions, is free from the extremes of moisture and of dryness, and that this point is to be found one-third down the sheet, and one-third across. In normal pulp, and especially in dry pulp, this system has several advantages, one of which is that the size of the triangles can be kept more uniform than is the case with strips taken from the centre of the sheet to the outside It would appear that this argument applies equally well to the long strips, but in practice it is not so easy to carry out the theory, and it has been found much easier to regulate the size of the single triangles than to retain the same degree of uniformity when samples are drawn by means of the triangle from the centre of the sheet.

Although this method of sampling has been so extensively used, yet it has been none the less open to the very serious objections that the middle of the sheet and the outer edge are entirely neglected Where the pulp is in abnormal condition, and where the edges of the bales are either wet or dry, then if a sample is cut from a point which ignores these two conditions of moisture, it seems only reasonable to suppose that error may be introduced, while, if the triangles be cut from the centre of the pulp to the outside, all the degrees of moisture contained in the sheet are represented in the sample, and, making allowance for all possibilities of error, the weight of evidence is in favour of this latter method, the general adoption of which would do much to remove obstacles in the way of attaining uniformity

The samples should be weighed off immediately During the sampling the triangles should be placed in a wrapper consisting of several sheets of pulp folded, or a tightly-stoppered bottle may be employed Where very large samples are to be drawn, a tin cylinder, fitted with a deep and closely-fitting lid, is a useful receptacle The bottle or tin is accurately tared, and the difference in weight is the weight of pulp contained

PULP-DRYING OVENS

In order to ascertain correctly the percentage of moisture contained in a sample of pulp, it is, of course, necessary that the drying operation should be conducted in a properly-constructed oven, which will maintain the required temperature for any length of time which may be necessary for the complete drying of the sample.

197. Pulp-drying ovens fall into three classes. (1) Of these, the first is the water oven, constructed on the lines of the ordinary laboratory drying oven, and described in paragraph 20. For the purposes of weighing, the pulp is taken out of the oven and first cooled in a suitable desiccator. (2) The Air Oven · This, also, is constructed on the usual lines (see paragraph 21), and the pulp is cooled before weighing (3) An oven wherein the weighing of the pulp is effected without removing the sample from the drying chamber.

198. The Water Oven.—The water oven, when used for pulp testing, consists of a copper drying chamber, surrounded by a water jacket, as described in paragraph 20. The drying chamber is fitted with perforated shelves, upon which the sample of pulp is spread out, and the steam escapes through an aperture in the top of the oven. In using this oven, the sample is packed loosely upon the shelves, and dried until it ceases to lose weight, when it is removed from the oven, placed in a suitable vessel, cooled under a desiccator, and weighed.

With such an oven as this, where the heat is supplied by a water jacket, the temperature of the drying chamber never exceeds 100° C., in fact, the average temperature obtained, even when the water in the jacket is kept at boiling point, is generally about 96° C. If we assume that it is absolutely necessary for pulp to be dried at 100° C., then the water oven does not fulfil the necessary conditions, for continued observations, with an oven measuring about six or seven inches square, indicated that 100° C. was never reached. The oven tested was an ordinary copper laboratory drying oven, jacketted with asbestos in order to prevent loss of heat, and the observations extended over several years, during which some hundreds of moisture tests of wood pulp were made. It may therefore be taken that, with such a water oven, the exact temperature of boiling water is not reached in the drying chamber.

The advantage of the water oven for pulp testing is, that a temperature of over 100° C. cannot possibly be attained. This is a very important consideration, and one which will be emphasised later. It will readily be seen that, when fitted with a condensing arrangement, together with a constant water feed, it is possible to leave the water oven without any attention for a considerable time, with the certainty that the specified temperature for pulp drying, 100° C., will not be exceeded.

199. The Air Oven.—This apparatus is constructed in several types. A common pattern is that of the ordinary laboratory hot-air oven, which consists of a square drying chamber of copper or sheet iron, with an air jacket, the heat being supplied by a Bunsen burner underneath, and the hot gases passing through the jacket. In other respects, the air oven is similar in construction to the water oven, the difference being that in the water oven the heat is derived from water in the jacket, kept at boiling point, while in the case of the air oven the heat is supplied by means of air heated by the burner, and circulating around the drying chamber.

It is a very easy matter, when using the air oven, to exceed 100° C in the drying chamber With careful regulation this temperature may be maintained, but, in any case, constant watching is necessary, and if the gas supply varies, it is not an infrequent occurrence to find that, although it has previously been regulated to 100° C., in a comparatively short time the thermometer will indicate perhaps 105° C Further, when using an air oven, assuming that, from variations in the gas supply, or from other reasons, the temperature of the drying chamber has been allowed to drop, it is a very natural expedient to raise the flame of the burner considerably, and then to forget to re-adjust it when the correct point has been reached When it is again noticed, the thermometer may possibly be ten or twenty degrees higher, the result is that often the sample of pulp is scorched or charred. It is, of course, possible, by the use of a gas regulator, to prevent such an occurrence, but in practice there are very few works laboratories which boast such a fitting to the pulp oven. Therefore, under ordinary circumstances, without very careful supervision, the drying chamber of a hot-air oven is very liable to attain a temperature of more than 100° C, and for this reason a water oven is infinitely preferable

200. Oven in which the sample is weighed in the Drying Chamber.—The third oven which we shall here consider is that pattern in which the weight of the dried pulp is ascertained without removing the sample from the drying chamber. The construction of this special form of oven is as follows: The drying chamber generally takes the form of a cylinder of copper or sheet iron, surrounded by a jacket, above the oven is arranged the weight beam, one end of which is fitted with a scale pan, while from the other end of the beam a rod is suspended, and passes through the cover of the drying chamber, and from this rod hangs a wire cage. The sample of wet pulp is placed in the cage, the temperature of the oven is raised by means of a ring burner underneath the air jacket, and the gases from the burner, together with the moisture from the pulp, escape through separate pipes leading into a flue. The temperature of the bath is adjusted to 100° C, and this temperature is maintained for some hours When it is assumed that the bulk of the moisture has been driven off, the rod carrying the cage is attached to one end of the weight beam, and counterpoised with weights upon the scale pan at the other end of the beam

It will thus be seen that the dry pulp is actually weighed at 100° C, without being removed from the drying chamber This oven being heated by hot air, the same objection holds good which has been mentioned in paragraph 199. The gas supply needs very careful regulation in order to maintain the temperature of the oven at 100° C, and it is not an uncommon occurrence with such a drying oven for this temperature to be exceeded It sometimes happens, as in the case of the ordinary hot-air oven, that the pulp is scorched by reason of the temperature having reached a point above 100° C The author has seen many samples which have been badly charred in such an oven The greatest objection, however, to this special type of apparatus is that the pulp is weighed at a temperature of 100° C., instead of at the temperature of the laboratory, as in the case of the two previously described ovens, in which the pulp, before being weighed off, is transferred to a dessicator and cooled to the temperature of the room in which the weighing operations are performed Again, this type of oven is open to the objection that the weight beam, which is suspended above the heated oven, is affected by the currents of hot air which rise from the oven In ordinary analysis, it is an invariable rule to cool down all substances to the temperature of the room by means of a desiccator, before weighing, the reason for this precaution being that when a heated substance is placed upon the scale pan of a sensitive balance, the current of hot air which rises from the hot body affects the beam, and introduces a very considerable error into the weighing operations If the weight beam used be at all sensitive—which, of course, should be the case—it is not difficult to understand the error In a word, this objection is that the pulp dried in such an oven, and weighed off at 100° C., will show a higher percentage of moisture than a duplicate sample dried in either the ordinary laboratory water oven or the ordinary hot-air oven, and cooled under the desiccator before weighing, and the ordinary rules of analysis apply, with equal force in the testing of pulp as in the testing of any other substance

With regard to the respective merits of the three types of oven here described, it may be pointed out that the water oven is perfectly safe as regards high temperatures, so long as the water supply is maintained, the temperature of the drying chamber cannot possibly reach a higher point than 100° C. This temperature is, however, rarely reached, but it is generally accepted that a sample of pulp dried in a water oven until the weight is constant, will give a perfectly accurate result. The hot-air bath, when carefully regulated, and the temperature maintained at exactly 100° C. is a perfectly satisfactory oven, the drawback here, however, being that the regulation of the gas supply, so as to maintain the exact temperature, is a somewhat difficult matter. When, however, an efficient gas regulator is fixed to this bath, there is no trouble with regard to high temperature. The special form of oven in which the pulp is weighed, without removing from the drying chamber, occupies a position upon which opinions vary It may be enough to say here that tests carefully conducted in this oven, and contrasted with tests on duplicate samples made in water ovens and hot-air ovens—in which tests the pulp was cooled before weighing—show from one to four per cent. more moisture, and these variations are far too serious to be admitted It would therefore appear that either a water oven or a hot-air oven will give the most satisfactory results, and accordingly it is recommended that one or other of these types should be adopted In practice, the water oven has been found to be the most satisfactory, since, as long as the water jacket contains any water at all, the temperature of the drying chamber is maintained at a fairly constant figure, which cannot possibly exceed 100° C. This type of oven is therefore recommended as being the most reliable for use in pulp testing.

201. The Use of the Desiccator—Although there is some controversy existent at present with regard to the proper condition in which pulp should be weighed off—that is, whether the weight should be taken at 100° C., or at the temperature of the laboratory—it may be taken that the generally accepted practice is to dry the pulp at 100° C., to cool by means of the desiccator, and to weigh at the temperature of the laboratory

Carefully conducted experiments have shown that the error introduced by weighing off dry pulp while warm is considerable, and this being so, it is far better to take the whole of the weighings after the pulp has been cooled. For use in pulp testing a large desiccator is, of course, necessary. For samples of moderate size such a desiccator is not difficult to procure, and, where very large samples are to be handled, a large tin cylinder, with a tightly-fitting lid, may be employed The hot pulp is put into the cylinder, the lid placed in position, a rubber band slipped over the joint, and the whole allowed to cool, and then weighed The pulp is afterwards taken out, and the tin, with the rubber band, is weighed, the difference in the two weights being the dried pulp

202. Calculation of Results—Moisture tests of wood pulp are thus expressed —

Total Moisture .	per cent
Bone-Dry Pulp	per cent.
= Air-dry Pulp	per cent

These figures are obtained as follows —

(1) Calculate the moisture from the loss in weight of the sample when dried at 100° C. = Total Moisture.

(2) Deduct this figure from 100 = Bone-dry Pulp.

(3.) Add one-ninth to the percentage of Bone-dry Pulp = Air-dry Pulp.

(4.) 50 per cent Air-dry Pulp—This is twice the Air-dry weight.

203. Air-Dry Pulp in Parcel.—The rule for estimating the weight of air-dry pulp in the parcel which has been tested is as follows. (1) Calculate weight in pounds of the bales sampled; (2) from this figure calculate the number of pounds in the whole parcel; (3) obtain the air-dry percentage figure; (4) multiply the gross number of pounds by the percentage of air-dry pulp, and divide by 100 The result is the total number of pounds of air-dry pulp contained in the parcel tested. These figures may, of course, be converted into tons, cwts., qrs., and lbs if required. If the weight of 50 per cent air-dry pulp is required, the air-dry figure is multiplied by 2

204. Uniformity in Sampling and Testing.—At the present time, considerable agitation is taking place amongst pulp buyers and sellers with regard to the great want of uniformity in the methods adopted for sampling and testing pulp. Much has been written upon this subject, but there still remain many differences of opinion between buyers and sellers, and also between the various public analysts engaged in the work of settling claims for excess moisture. It is very desirable, therefore, that uniform methods should be adopted, not merely of sampling, but also of testing, and this uniformity should extend to the apparatus to be employed in the testing. The determination of the moisture in a parcel of pulp presents difficulties, it is true; but these difficulties are not such as to render impossible the task of devising uniform methods for carrying out the various operations. The suggestions here made are simply the embodiment of considerable experience in pulp sampling and testing, and the adoption of some such system would do much to remove discrepancies in results, and to secure some measure of uniformity in the sampling and testing of wood pulp.

205 General Rules to be observed in Sampling and Testing Pulp with a view to securing uniformity of results.—

(1) *Percentage of Bales.*—The percentage of bales to be sampled to be 2 per cent. of the whole.

(2) *Heavy Bales*—In 2 cwt. bales, no bale weighing more than 2 cwt. 1 qr to be sampled. In 2½ cwt. bales, no bale weighing more than 2 cwt. 3 qrs. to be sampled. In 4 cwt. bales, no bale weighing more than 4 cwt. 1 qr. to be sampled.

(3.) *Light Bales.*—Light bales, if perfectly sound and intact, to be included.

(4.) *Damaged Parcels.*—To be dealt with as indicated in paragraph 193

(5) *Method of Cutting Samples.*—Samples to be drawn by cutting two triangles from each sheet, one from the centre to the end of the bale, the other from the centre to the side of the bale, the base of the triangle to measure about 1 inch; samples to be uniform in size.

(6.) *Number of Sheets per Bale.*—Four sheets per bale to be taken for the sample. (In accordance with the present contract note three sheets only per bale are drawn.)

(7.) *Pulp Drying Oven*.—A water oven, or a hot-air oven fitted with a gas regulator, to be used for drying the sample.

(8.) *Weighing off the Dried Sample*.—The pulp, when dry, to be cooled before weighing. Samples to be weighed to the tenth part of a gramme.

(9.) *The Metric System*.—Metric weights should be used in weighing samples, and a balance which will carry 3,000 gm., and turn with 0·1 gm. should be provided.

CHAPTER VIII.

Water Analysis, Purification, Filtration, and Softening.

The subject of water analysis is one which should have considerable interest for the manufacturer. He uses vast quantities of water daily in many of his operations, and it would often be very convenient to have at hand the requisite information for carrying out the different tests necessary for the proper control, not only of the water supply for process operations, but also for the examination of water used for steam raising Added to this is the urgent necessity for keeping a close watch on the effluent which is turned into the river or watercourse. In these days no manufacturer should be satisfied with the test of the river inspector; he should have an accurate record of the daily condition of his effluent, and such a record could not fail to be of infinite service and guidance on many occasions This question of effluent testing, however, will be dealt with in the following chapter.

It is not necessary, for trade purposes, to make a full analysis of every sample of water submitted for examination. Generally speaking, two or three estimations are all that are required; but it is proposed in this chapter to give full instructions for carrying out the different estimations required for a fairly complete water analysis, and the operator can then select those tests which are required in any particular case.

Before proceeding to deal in detail with the different separate estimations, it will be useful to preface the working instructions with a few general remarks on the subject of water analysis.

Water analysis involves very exact and careful manipulation in the carrying out of the different operations, which at the same time are exceedingly simple, and only require practice to be executed with sufficient accuracy for the purpose of the manufacturer. The apparatus used for water analysis must not be employed for any other test; this is a rule which should be strictly observed. The retort, measuring flask, graduated jar, pipettes, etc., should be set aside for the purposes of water analysis, and care should be taken that they are not used indiscriminately for any other purpose. This rule has, perhaps, one exception, and that is with regard to the platinum apparatus In water analysis, a platinum evaporating basin should be used, and, although platinum is expensive, yet the accuracy and convenience of working which it offers, are sufficient justification for the additional expense, as a platinum basin is useful for many other operations. With this exception, apparatus should be set apart for water analysis, and retained for that specific purpose.

Absolute cleanliness must be observed in working on waters While the tests are being made, ammonia and acid gases must not be generated in the laboratory, else the results will often be vitiated, especially when the distillations for ammonia and albumenoid ammonia are proceeding. When a piece of apparatus is about to be used, it must be thoroughly cleaned. If it has previously been employed for any other test, it should be cleaned with a little sulphuric acid, and thoroughly washed, using tap water plentifully for rinsing purposes

206 Ammonia-Free Water.—In water analysis, an ample supply of distilled water is a necessity, and, further, water which is free from ammonia is required for the determination of the ammonia-free and albumenoid Ammonia-free distilled water is not easy to obtain, but it may be prepared in the following way Clean a large stoppered retort, with a capacity of about a litre and a half. Introduce a little strong hydrochloric acid, and shake the liquid well round the sides of the retort, finally pouring it out through the tubulure. Now rinse well with tap water, until the last few drops have no acid taste The retort may now be regarded as clean

Fit up the retort in a heavy stand, and connect up to a Liebig condenser. A strong clamp must be used to grip the retort, and the clamp should be packed with a little newspaper. The best way of securely clamping a water retort in its stand is to take a strip of newspaper of some length, and fold it into a narrow strip, which will just cover the arms of the clamp; now fold again, so as to produce a square wad of paper, which covers one of the arms. Two of these wads are required; a ring is fitted on the stand in order to support the retort, and the clamp is adjusted so that the arms grip the neck tightly Care must be taken in making this adjustment, and the clamp must be tightened very gradually. In the text-books on water analysis, instructions are given *not* to use a ring as a support for the retort, but in practice it will be found a considerable convenience, especially when the operator is not accustomed to water analysis.

Having fitted up the retort, ensure that the cooling-water supply and waste connections are in order The cooling-water must run in at the top of the condenser—that is, next to the retort, and flow from the bottom. A rose Bunsen burner should be used, of a good size, and it should be employed with the rose in position.

Now introduce about 800 c.c of tap water into the retort—or about two-thirds full. Fill the stopper in tightly, and wipe off any moisture from the bottom and sides of the retort. Light the burner, and turn the flame down very low—as low as it will go without "lighting back." Place the burner under the retort, down on the bench, and not close under the retort bottom. Allow the retort to warm up in this way, wiping off the moisture as it condenses. The condenser must be inclined at such an angle that it will permit a small flask to be placed under the end of the tube to receive the distillate, and this will mean that the burner will have to be packed with blocks to bring it close up to the retort. Three blocks of wood, of different thicknesses, should be used for this purpose, and by this means the burner may be raised, by degrees, after the retort has warmed up, until it fits close underneath. Now turn on the gas fully, see that the condensing water is flowing steadily, and the water will presently be distilling at a fairly rapid rate.

If the condenser has been used for other purposes, and is dirty, the best way of cleaning is to steam it, by keeping the cooling water running very slowly, until faint puffs of steam make their appearance at the end of the condensing tube. This steaming must be very carefully performed. It is enough that the distillate comes through quite hot, and a little steam appears occasionally. Too much steaming will soften the rubber joints of the condenser; but in some cases of filthy condensers the only possible way of cleansing them is by steaming for some time.

After about 500 c.c. of water have distilled over, fill a Nessler tube, holding 50 c.c., with the distillate. Introduce 2 c.c of the Nessler reagent (see "Nesslerising," paragraph 223), and observe the colour. Fill another tube with tap water, and place the two on a white porcelain plate for comparing the colours If the distillate develops a brown or yellowish tinge, the retort and condenser are still dirty, and the distillation must be carried on It is quite possible that the first charge of water will not yield a distillate which remains colourless after the addition of the Nessler reagent. If this is the case, the distillation may be continued until the water gets down to just above the ring, when it must be stopped

The distillate resulting from this cleaning of the retort and condenser is ordinary distilled water, and is ready for use for laboratory purposes Empty the retort, fill again with fresh water—which may have been previously boiled in a clean flask—and continue the distillation, testing frequently with the Nessler reagent After a time a tube will show no colouration when compared with a tube of tap water The retort and condenser are now clean, and a quantity of ammonia-free water can be obtained from the retort.

The distilled water which has been obtained during the cleaning of the apparatus, should be bottled and labelled as ordinary distilled water, the ammonia-free water should be kept in a tightly-stoppered bottle, and labelled as such. About 500 c.c of this water may be obtained from a charge after thoroughly cleaning the apparatus, and a retort, when once yielding free water, should be kept constantly producing it until a good supply is accumulated, the same process being repeated in the distillation; first the retort is filled with tap water, and distilled until free from ammonia, then the distillation is continued until the contents of the retort are just above the supporting ring, when the distillation is stopped, and the retort re-started with a fresh charge of tap water.

If ammonia-free water is kept in tightly-stoppered bottles, it will keep perfectly free from ammonia for a considerable time, but on each occasion when it is used, a test should first be made with Nessler reagent to confirm its suitability for Nesslerising purposes.

207. **Total Solids.**—Clean, dry, and weigh two platinum basins, each holding about 100 c.c Shake the sample of water well. Rinse a 70 c c. pipette with tap water, and empty Suck up a little of the shaken sample into the pipette, rinse round well, and run out. Now fill up to about an inch above the mark on the stem, and close tightly with the end of the finger. Carefully release the pressure, and empty drop by drop until just to the mark. Run into the weighed dish. Place the dishes containing the two lots of 70 c.c. of sample on the water bath, and evaporate to dryness; transfer to the water oven, and weigh at intervals until the weight is constant. The increase of weight of the dish is the amount of total solid matter contained in 70 c c of the sample. The weights of the dishes should be carefully ascertained before testing, and the weighings made to the third place of decimals. The weight of the residue, multiplied by 1,000, gives the number of grains per gallon of total solids—thus, if from 70 c.c. of water, 0·06 gm of residue is obtained, then 0·06 × 1000 = 60·00 grains per gallon.

208. **Suspended Matter**—Dry two filter papers in the water oven. Before drying, fold the papers just as for use in filtering, and then fold again, so that the folded paper will fit into a weighing bottle. Open the papers out before drying Two bottles, cleaned, should be put into the oven with the papers, first removing the stoppers from the bottles. After about an hour the papers will be dry. Less time than this may suffice, and the papers may be weighed at intervals of half an hour until the weight is constant. In weighing, place the papers, folded, into the bottles, while still *inside the oven*, fit the stoppers, and place under the desiccator to cool. Weigh, and replace in the oven. After half an hour, weigh again, and, if constant, proceed with the test. The two weighing bottles should be marked, 1 and 2, with a file, and the filter papers marked in pencil on the margin to correspond.

Fit the papers in funnels. Weigh off two platinum basins holding about 100 c.c. Take the bottle containing the sample of water, and shake well. Measure off two lots of 70 c.c. of the water, and pass through the filters, catching the filtrate in the dishes. Wash the filters, rinsing out the measures with distilled water, and allow the washing to run through the filters. Allow to drain, and then place the funnels, with filter papers untouched, in an upright position in the water oven, and dry. When dry, take from the funnel, and very carefully fold as when previously weighed, taking care not to break the dry paper, cool, weigh off, dry again for half an hour, cool, and again weigh The difference in weight in the two weighings indicates the suspended solids contained in 70 c c of the water, and this, multiplied by 1,000, gives the number of grains per gallon.

209. Dissolved Solids—Filter a quantity of the sample through a filter paper Clean, dry, and weigh two basins. Into each pipette off 70 c c of the filtered sample. Evaporate to dryness on the water bath, finish off in the water oven, and weigh at intervals until constant The gain in weight indicates the dissolved solids in 70 c c of the sample Multiply this by 1,000, and the result is the number of grains per gallon.

210 Loss on Ignition.—This is a useful approximate method of estimating the amount of organic matter in an effluent containing much fibrous material, or other organic constituent. The total organic matter is determined by taking the dried residue from the "total solids" Ignite this residue carefully over a Bunsen burner, or at the mouth of the muffle furnace, heating gently up to faint redness, cool, and weigh. Heat again, cool, and weigh. The weighings should be continued until constant. The result is the total organic matter.

Tests of organic matter by means of the "Loss of Ignition" test are not absolutely correct, as in some instances volatile salts may be driven off, but for the purposes of works testing, where approximate results only are generally required, the tests are very useful indicators of the amount of organic pollution which may exist. On the other hand, "Loss on Ignition" tests afford a very convenient method of controlling the amount of fibrous matter running into the river in a paper mill effluent, for instance Such fibrous matter is not only a polluting agent, but it is also a very serious loss to the paper maker. By the use of plant specially designed for the purpose, the loss may be averted, and, at the same time, the pollution from this source very largely diminished.

211 Total Hardness—Take an eight-ounce medicine bottle, and fit it with a clean, *new* cork. Pipette off 70 c c of distilled water into the bottle. Fill a Bink's burette with the standard soap solution (see paragraph 58). A Mohr's burette will, of course, answer quite as well Run in half a c c of the soap solution, and shake well, watching to see if a lather forms Run in another half c c., and again shake A lather should now form, which should remain on standing for four minutes, with the bottle placed on its narrow side Seventy c.c of distilled water should not require more than 1 c c of the standard soap solution to procure a permanent lather; if more is required, it is an indication that the bottle, or the cork, is dirty

Now empty the bottle, and rinse out with tap water Measure off 70 c.c of the sample into the bottle A convenient measure is a 100 c c. jar, graduated in c c 's Fill up the burette to the zero mark with the soap solution, and run two or three c.c, into the test bottle. Shake well. Continue adding the soap solution until a lather begins to form, when the addition should be continued in smaller quantities of about 1 c c each time, shaking thoroughly after each addition. When a lather forms, continue the agitation for some time; lay the bottle on its side on the bench, and watch If the lather breaks, the test is not finished, and more soap solution must be added When the lather remains permanent, and does not break after standing for four minutes, the test is complete Read off the number of c c. of soap solution required to form the lather Assuming that this is 13 c.c , then the total hardness of the sample is 13° In the estimation of total hardness, if the number of c c of soap solution exceed 17, 70 c c should be diluted with 70 c c of distilled water, and 70 c c of this mixture titrated The number of c.c. now consumed is multiplied by 2, and 1 c.c deducted for the distilled water. The result is the total hardness of the water. For instance, 70 c.c. of sample was diluted with 70 c c. of distilled water, and 70 c.c. titrated, which required 10 c c of soap solution. Then, 10 × 2 = 20 − 1 = 19° total hardness

212. Permanent Hardness—Take 70 c c. of the sample, and dilute with 70 c.c. distilled water. Place the 140 c.c in a small flask or conical beaker, and boil down to about the original bulk. Filter, and wash so that the filtrate and washings measure 70 c.c Titrate this with soap solution, and deduct 1 c.c for the distilled water. The result is the permanent hardness. Thus, 70 c c. sample were diluted with 70 c.c. distilled water, boiled, filtered, and washed up to 70 c.c.; the filtrate took 8 c c. soap solution. Then 8 − 1 = 7° permanent hardness. In the case of a very hard water, where the soap solution required is above 17 c.c., the sample must be diluted with distilled water as before, taking, for each 70 c.c. of sample, either one, two, or more equal volumes of distilled water. Thus, 70 c.c. of a water were diluted with 140 c.c. of distilled water; 70 c.c. were boiled, filtered, and washed as above. Nine c.c. soap solution were required to form a lather. Then 9 × 3 = 27 − 3 = 24° permanent hardness.

213. Temporary Hardness.—This is the difference between the total hardness and the permanent hardness. Thus, a sample shown 26° total hardness, and 8° permanent hardness, then the temporary hardness (that is, hardness which may be removed by boiling) = 16°.

Note.—Hardness tests should always be made in duplicate.

214. Chlorine.—Pipette off 70 c.c. of the sample into a clean, white porcelain evaporating basin (a basin should be reserved for this test). Test with a litmus paper; if the sample reddens blue litmus paper, neutralise the acidity with a little pure carbonate of magnesia; if the water is alkaline, add a drop or two of pure nitric acid, until neutral. In any case, the water must be neutral before proceeding with the test. This neutralizing can be accomplished by adding small quantities of pure magnesium carbonate and pure nitric acid until the neutralization is effected.

Fill a 10 c.c. burette (graduated in tenths of a c.c., and provided with a glass stopcock), with solution of silver nitrate containing 1.7 gm. nitrate per litre. Take the 70 c.c. of water in the porcelain basin, and add two drops of a solution of pure bichromate of potassium; this will colour the water yellow. Now add the silver solution from the burette, drop by drop, until a reddish precipitate is formed, which remains on stirring. This is the end-point of the re-action. It is unmistakable, but if any doubt be felt as to the colour, take 70 c.c. of tap water, add two drops of the indicator, and stand side by side with the water being tested. The change in colour is then easily compared by glancing at the blank sample. Repeat the test.

In testing waters and effluents for chlorine, it is well to make two estimations, one on the water itself, and the other on the ignited residue from the "Total Solids." The latter is the more reliable test, but often the chlorine is required quickly, and in such a case the test of the water itself is a convenient method. In the estimation of the chlorine in the residue, take the dish containing the ignited residue, add a little distilled water, and rub off the sediment from the sides of the dish with a rubber-tipped glass rod. Wash the contents of the dish into a white porcelain evaporating basin, neutralise as above, and proceed as there instructed.

The calculation of the chlorine present in the sample is then made as follows, assuming that 70 c.c. of the sample required 2 c.c. of centi-normal silver solution:—

70 c.c. water = 2 c.c. $\frac{n}{100}$ silver nitrate.
70,000 „ = 2,000 c.c. „ „
1 c.c. $\frac{n}{100}$ silver nitrate = 0·000355 gm. chlorine ; then
2,000 × 0·000355 = ·71 grains chlorine per gallon.

215. Free Ammonia.—Having cleaned the retort and condenser as described in paragraph 206, measure off 700 c.c. of the sample carefully, and introduce into the retort by means of a large funnel. Before running in the water, place in the retort about 2 gm. of freshly-ignited anhydrous sodium carbonate. Replace the stopper in the tubulure, and bring to boiling. Catch the first 200 c.c. of distillate in a stoppered flask, and place on one side. Then take off 50 c.c. in a Nessler glass, and Nesslerise as instructed in paragraph 223. If ammonia is still present, a second and third tube must be collected, until the last tube shows no colouration with the Nessler reagent Then stop the distillation. Mix the contents of the 200 c.c. flask containing the first part of the distillate, and also Nesslerise, adding the result to that obtained from the separate tubes.

216. Albumenoid Ammonia.—To the contents of the retort, after the distillation of the free ammonia, add 300 c.c. of ammonia-free water, prepared as already indicated. Take 50 c.c. of alkaline permanganate solution (paragraph 57), with 50 c.c. of ammonia-free water, and boil down to 50 c.c. Add this to the contents of the retort. Now take a piece of soft glass tubing, and draw it out in an ordinary gas flame, so as to form capillary tubing of very small bore. Break this capillary tubing into pieces half an inch long, seal up one end in the gas flame, and place a dozen of the tubes in the retort Heat up again, and take off 200 c c of distillate. Then collect separate tubes, as in the case of the free ammonia, Nesslerising each tube separately, until a tube shows no colouration with the Nessler reagent Add the results of the final Nesslerising to the sum of the separate tubes, and the result will be the albumenoid ammonia contained in 700 c.c. of sample.

In conducting the distillation for albumenoid ammonia, it is very necessary to avoid bumping When the contents of the retort get low, bumping is very common, and it is not at all unusual for an experienced operator to find the contents of the retort come seething over through the condenser. This may be avoided by adding the capillary tubes as indicated above, and by gently rocking the retort when a burst seems imminent. With very bad waters, it may be necessary to continue rocking throughout the whole of the latter part of the distillation. With practice, however, a patient operator will not have many accidents, but great care is absolutely necessary, especially when the water being distilled is an exceptionally hard one.

The method of calculating the ammonia will be found under "Nesslerising," paragraph 223.

217. Iron in Solution.—Evaporate 70 c.c of the water to dryness in a platinum basin over the water bath; dry in the water oven, and ignite to dull redness over a Bunsen burner or in the muffle furnace (The residue thus obtained corresponds to that from the "Loss on Ignition," paragraph 210.) Now add 1 c c of a mixture of equal parts of *pure* nitric acid (free from iron) and distilled water, and evaporate to dryness over the water bath Take up the residue from this second evaporation with 1 c c. of a mixture of pure hydrochloric acid in 10 parts of water, add distilled water, and pour into a Nessler tube; wash the basin well (the washings going into the tube), and make up with water to the 50 c.c. mark. Into another Nessler tube place 10 c.c. of the standard iron solution (paragraph 61), add 1 c.c. of 10 per cent. hydrochloric acid, and dilute to the 50 c.c. mark. To each tube now add 1 c c of a 4 per cent solution of potassium ferrocyanide, which will cause a blue colour to develop The tube containing the standard iron solution must now be made to match the water tube; if the colour produced by 10 c.c. of iron standard is too faint, try another test with 20 c.c., and so repeat until the exact colour is produced. Then, assuming that 10 c.c of standard has been required, and each c c. is equal to 0·00001 gm. of metallic iron, then

70 c.c water = 10 c.c iron solution.
70,000 c.c. of water = 10,000 iron solution.
10,000 × 0·00001 = 0·10 grain of metallic iron per gallon

The above method for the estimation of iron in water is given in "The River Irwell and its Tributaries," by Davis Bros.

218. Silica.—Measure off 700 c c. of the water; if a dirty sample, it must be filtered. Add a few drops of pure hydrochloric acid to render the water distinctly acid

Weigh off a clean platinum dish, and in this evaporate the 700 c.c. to dryness. Keep the measured water in a stoppered litre flask, and see that the platinum dish is kept filled. The evaporation should be carried out over the water bath

When completely evaporated, transfer to the water oven, and dry. Moisten the residue with a few drops of pure hydrochloric acid, and add about 50 c.c of distilled water Rub off the residue from the sides of the dish with a glass rod tipped with rubber, and filter Wash the residue on the filter paper, dry, and weigh; the ignited residue, multiplied by 100, gives the grains per gallon of silica present in the water

219. Soluble Lime.—This is estimated in the filtrate from silica; add ammonium hydrate; filter off the precipitate (iron and alumina), and to the filtrate add oxalate of ammonia solution, this precipitates the lime as oxalate of lime Warm, allow to stand for a few hours, and filter through a small filter; wash well, dry the precipitate, and gently ignite in a platinum crucible or dish This ignition converts the precipitate into carbonate of lime. After heating gently, allow the precipitate to cool, and moisten it with water in which a piece of carbonate of ammonia has been dissolved. Ignite again gently to approaching redness, cool in the desiccator, and weigh the oxide of lime. The weight, multiplied by 100, gives the grains per gallon of soluble lime.

220. Soluble Magnesia.—The filtrate from the lime contains the magnesia. To this filtrate add a little strong nitric acid, and evaporate to small bulk, add a little chloride of ammonium solution, an excess of ammonium hydrate, and then phospate of soda solution, also in excess. This precipitates the magnesia as magnesium pyrophosphate Allow to stand all night (or 12 hours), and filter through a *small* filter paper, wash the precipitate with weak ammonia water, dry, ignite the precipitate, cool under the desiccator, and weigh when cool

The weight of magnesium pyrophosphate so obtained, multiplied by 100, and then by 0 36036, gives the number of grains per gallon of magnesia (magnesium oxide) contained in the water.

221. Lead—The following is a quick and fairly reliable test for the detection of lead, iron, and copper in water Place 70 c c. of the water in a white porcelain basin, and add a little ammonia; dip a clean glass rod into a solution of ammonium sulphide, and stir the water well with the rod. If a dark colour or precipitate results, the presence of iron, lead, or copper is indicated. Make the solution acid by means of a few drops of pure hydrochloric acid If the colour disappears, iron is present If the colour remains, copper or lead is present.

222 Copper.—Copper may be detected as follows· Separately evaporate a quantity of the water to dryness, acidify with hydrochloric acid, and pass sulphuretted hydrogen gas through the liquid. If a precipitate forms, filter it off on to a small filter paper, and dry. Place the filter in a platinum crucible, ignite over a Bunsen burner, and add a drop of strong pure sulphuric acid, which will form copper sulphate Cool, and add a little water, then a few drops of ammonia, and a blue colour will result.

NESSLERISING

223. The estimation of minute quantities of ammonia in water is effected by means of Nessler's reagent (paragraph 60). The distillate containing the ammonia is obtained as described in paragraphs 215 and 216. The process of Nesslerising is conducted as follows·—

Free Ammonia.—This is generally contained in the 200 c.c. of distillate caught in paragraph 216, but the distillation is continued to ensure accuracy. Take the first tube after the 200 c.c has distilled, and add 2 c c. of Nessler reagent. If ammonia is present, a faint brown or yellowish colour will develop Take 50 c c. ammonia-free water (paragraph 206) and to this also add 2 c.c. Nessler reagent. If no difference in colour is noticed, the 50 c.c. of distillate is free from ammonia. If, however, a colour is formed, take a *clean* Nessler glass, and in it place 1 c.c. of standard ammonia solution (paragraph 56), fill up to the mark with ammonia-free water, and add 2 c c. Nessler reagent, when a yellowish colour will appear. Stand the two tubes on a sheet of white paper, or a white porcelain tile, and compare the colours If the standard is too light, make up another test, using 2 c.c of standard ammonia, and so proceed until the two tubes are identical in colour. Note the number of c c. ammonia required to effect this. This is the operation of "Nesslerising."

As described in paragraph 215 and 216, in ammonia estimation the distillation is continued until 50 c.c. distillate show no colour (or a colour which is less than that produced with 1 c c. standard ammonia), the value of each tube in c c. standard being estimated as here described Assume that tube No. 1 took 2 c.c. standard, No 2 took 1 c.c., and No. 3 was free Now proceed to the 200 c.c. caught in the flask.

Mix thoroughly, measure off 50 c.c., and Nesslerise, making at least two tests; assume that 50 c.c. = 4 c.c. standard ammonia solution, then 200 c.c. = 16 c.c. standard, to which is to be added tubes 1 and 2 (3 c.c.), making 19 c.c. in all. This represents 700 c.c. water; therefore 70,000 (= a gallon) would require 1,900 c.c. standard, each c.c. of which = 0·00001 c.c. ammonia, so that 1,900 × 0·00001 = 0·019 grains of free ammonia per gallon.

Albumenoid Ammonia.—The Nesslerising is carried out in exactly the same manner as in the case of the free ammonia. The 200 c.c. of distillate first taken off are left till the last. Each subsequent 50 c.c. tube is tested until the distillate is free (or shows a colour equal to less than 1 c.c. of the standard), the number of c.c. of standard required by each tube being noted. Assume that three tubes of distillate are taken off after the

200 c c flask has been collected, and that No. 1 takes 4 c.c., No 2, 2 c c, and that No 3 is free Now mix the contents of the flask well, measure off 50 c c., and test with Nessler reagent; 8 c c of standard are required to match the colour; check this by a second test, then

50 c.c = 8 c.c standard.
200 c c = 32 c.c standard + 6 c c. (tubes 1 and 2) = 38 c.c.
38 c c. standard = 700 c c. sample.
3,800 c c. standard = 70,000 c.c. sample.

and 3,800 × 0·00001 = 0 038 grains per gallon of albumenoid ammonia contained in the sample.

Note—In Nesslerising operations, all apparatus must be scrupulously clean, and only ammonia-free water (see paragraph 206) must be used

224 Water Purification and Filtration—The impurities contained in water supplies may be estimated as described in the earlier parts of this chapter With regard to the filtration of water, there are many systems by which this may be accomplished For use in works where a large and continuous supply is a necessity, the Riddell filter, which is made and supplied by Messrs L. Hugh Bristowe and Co., of 47, Victoria Street, Westminster, London, S W., may be described as one which is in use in many large mills for purifying and filtering river water for manufacturing purposes It is well known that the river Irwell is by no means an ideal source of supply, but at the present time some four millions of gallons of Irwell water are being treated daily by this system, so that the Riddell apparatus may be regarded as fulfilling the requirements both of purification and filtration. Fig 36 shows a sectional view of the apparatus. The water is brought to the filter by the pipe B, and enters through the valve C, direct to the top of the filtering material D; passing through the filtering material, it is collected by means of strainers E at the bottom, and discharged through the clean water pipe F; at G is the overflow, through which the dirty water passes to the sewer during the cleaning of the filtering material. The radial arms H are attached to the hollow hub J at the lower end of the pipe K, which has a piston L at its upper end. The pipe and piston are free to move up and down in the cylinder M.

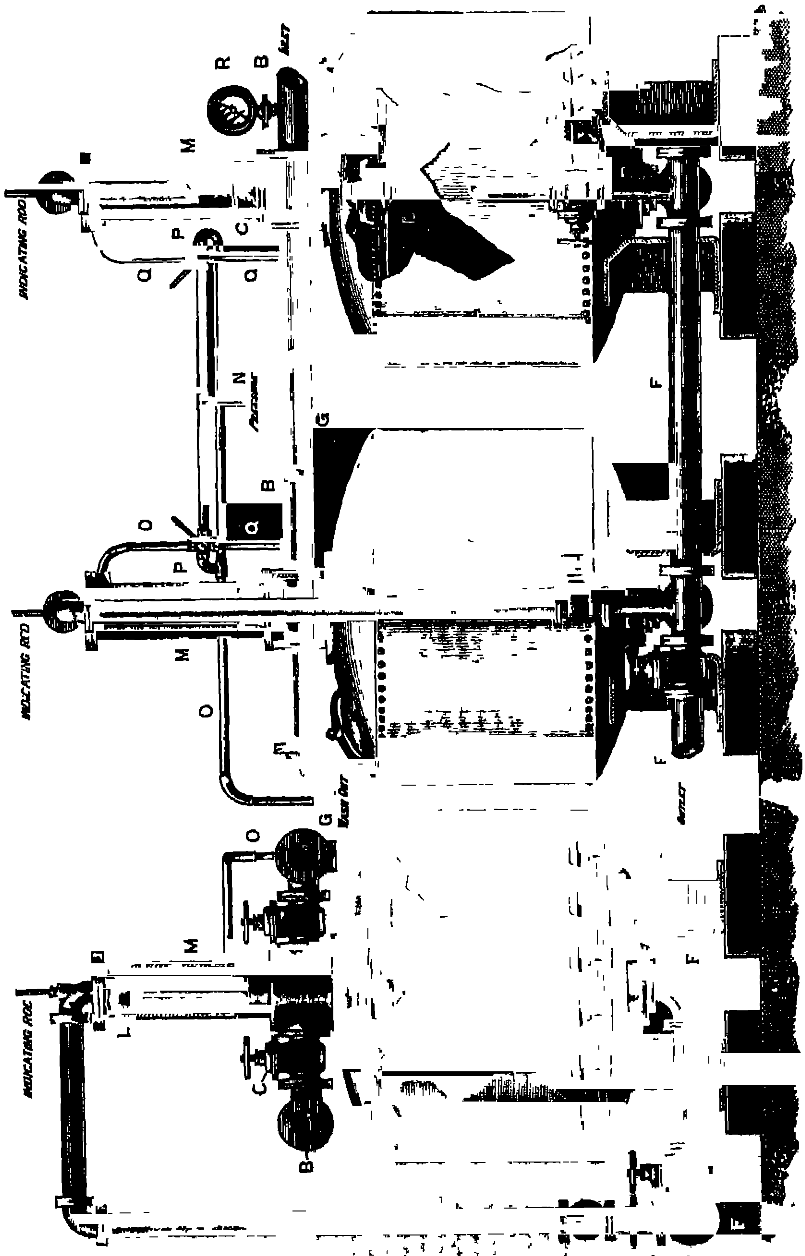


FIG. 96.—THE RIDDELL FILTER, SECTIONAL VIEW

The pipe N is connected with the boiler feed pump, the delivery main, or other source of pressure, and O is an exhaust pipe connected with the waste pipe By means of the four-way cock P, and the pipe O, the pressure may be conveyed to either side of the piston, as desired, while at the same time it opens up the other side of the exhaust and waste pipe O By moving the cock the pressure and exhaust are connected with the opposite sides of the piston.

In the regular work of filtering, the positions of the various parts of the filter are as shown, with the waste or overflow valve G closed, and the clear water valve open. A gauge R is fixed on the supply pipe, and gives the actual pressure, thus indicating the amount of resistance of the filtering material

The process of filtering proceeds steadily until the gauge indicates, by the resistance of the filtering material, that it is necessary to wash it. The period at which this will occur must naturally depend upon the nature of the water under treatment.

In plants of more than one compartment, the filter supplies itself with clean water for washing purposes No separate pump or tank need therefore be provided, as the one compartment which requires washing can be supplied with clean water from the other compartments The simplicity, combined with perfect efficiency of the washing operation in the Riddell system, is one of its chief merits.

The inlet valve of the compartment to be washed is closed, and the wash-out valve opened, with the result that part of the filtered water from the other compartments is forced upwards through the bed, putting the whole of it in suspension; as soon as the bed is thoroughly agitated, clean water is turned on through the radial arms, while at the same time they move up and down the compartment, thereby completing the agitation of the bed, and thoroughly scouring every particle of it.

The time occupied in this operation will, of course, vary considerably. It depends on two things; first, the nature of the water or effluent being treated; secondly, the available pumping pressure.

The supply of the chemicals necessary for the purification of the water is automatically regulated, and, when the proportion has been determined, and the apparatus adjusted, no further attention is required other than maintaining the supply in the reagent tank.

It is important that the pump supplying these filters should be constructed in such a manner as to ensure a steady supply to the filters while at work, and also that the pump should be capable of being speeded up during the process of washing

WATER SOFTENING.

225. Water Softening.—Hardness in water is due to the presence of salts of lime and magnesia. The object of all systems for water softening is the removal of these salts. Clark's process consists in the addition of lime water to the hard water, when, the carbon dioxide being removed, the lime and magnesia are precipitated.

The Archbutt-Deeley process, plant for which is made by Messrs Mather and Platt, consists in treating the water in a tank with a mixture of carbonate of soda solution and lime; the charge is then blown with air, the precipitate allowed to settle, and the clear tested water drawn off for use.

The "Carrod" system is another method of purification and filtration by the addition of lime and soda. This plant also is erected by Messrs. Bristowe and Co.

The "Carrod" patent water softener and purifier is automatic in its action; the opening and closing of a cock starts or stops the machine. The reagents are mixed in store tanks placed above the clarification tank; the tanks being in duplicate, the mixture in one is prepared while the other is in use, to prevent the continuity of the process being interrupted. The water to be softened is admitted to the clarification tank by a mixing trough, the proper proportion of the reagents passing in at the same time, and well mixing with it; the amount of each is determined by the size of the nozzles, which is kept constant by causing the feed in each case to pass through regulating tanks which are fitted with ball valves, thus ensuring the same head of water at all times. The water, after passing through the mixing trough, enters the clarification tank by means of a pipe carried to the bottom, then passes upwards, and by the time the water reaches the outlet it is rendered perfectly soft, clear, and fit for immediate use.

The softening of water should be controlled by means of the tests given in paragraphs 212 and 213. The amount of reagents used should be carefully watched, and, by means of the analytical data thus obtained, the efficient and economical working of the water-softening plant, whatever system may be adopted, is easily ensured.

CHAPTER IX.

Trade Effluents.

The testing and control of manufacturing effluents is a matter which is neglected, to a very great extent, by the person who, from a commercial point of view, is most interested—the manufacturer himself The daily press reports at regular intervals the proceedings of the river authorities, giving long lists of manufacturers who are to be proceeded against for pollution of the water courses. Generally speaking, the manufacturer occupies a somewhat helpless position, and the result is that in many cases he is harassed to a far greater extent than would be the case if he were to devote a little more attention to the establishment of proper control over the effluent which he turns into the river or stream.

Under existing circumstances, it generally happens that the River Inspector makes his appearance when he is least expected, and takes a sample of the waste water running away from the works, leaving a duplicate sample in the hands of the manufacturer. After an interval, a copy of the analysis of this sample is forwarded to the manufacturer. If the impurities contained in the effluent reach a certain point, the next step is that the manufacturer receives a notice from the Rivers Committee, requesting him to attend a meeting of that authority, before which tribunal he is invited to "show cause," why he should not take immediate steps to prevent a recurrence of the offence. If this is a first appearance, he is probably dismissed with a caution, on promising that it shall not occur again. If, however, he has been previously convicted, a substantial fine may be imposed, and the victim goes on his way chafing at his helplessness in the hands of the adamantine Commissioners.

There are several important points which may be usefully considered in connection with the state of things indicated above In the first place, the manufacturer has generally no means of checking the accuracy of the evidence upon which he is convicted and fined The River Inspector has perhaps been visiting his works for twelve months, and has periodically been taking samples of his effluent Reports of analysis of these samples of effluent have been sent to the manufacturer After several questionable effluents have been sampled, the proceedings follow which result in a substantial fine

In the first place, an absolutely independent control should be organised over the waste liquors turned into the river It is not sufficient to have periodical reports from a body, the object of whose existence is the detection of pollution. The manufacturer should have *independent* tests regularly made of the effluent which runs away from his works. If he is committing pollution, he will quickly be made aware of the fact, and can at once take steps to remedy the matter, but if he waits until the Inspector appears, just at the time when a bad effluent is running off, he may be convicted upon the worst sample which he has produced for a month The first step, then, which the manufacturer ought to take for his own protection, is that of establishing independent and reliable control of his effluents This is by no means a difficult matter to accomplish. The testing of trade effluents against the river authorities is a comparatively simple operation, and if the tests, which will presently be indicated, are applied, the manufacturer will accumulate a mass of evidence which, in many instances, would be invaluable when he is called upon to defend his actions before the river authorities.

In order to secure a record of the condition of trade effluents, it is not necessary that samples should be sent daily to a public analyst The objection to constant testing on the part of manufacturers is generally on the score of expense. They say, and with reason, that if they are to send samples daily to a public analyst, and are charged a guinea or half a guinea for a test, they cannot afford the cost A very simple way out of this difficulty would be to entrust the simple tests here described to the hands of an intelligent person—not necessarily a trained chemist, but with some knowledge of chemistry—who, with a little practice, will be quite competent to [illegible] tests necessary for the establishment of an approximate check upon the quality of the effluents

It is, of course, imperative that representative samples should be drawn. If the effluent is of uniform composition throughout the day, then one sample is sufficient to represent the day's output, but a good plan is to take a sample several times a day, mixing thoroughly, so as to obtain an average result A test of this average sample will give a very fair record as to the quality of the effluent for the day In the case of a paper mill, for example, samples might be drawn at four different times during the day, say at nine in the morning, twelve noon, three in the afternoon, and six o'clock in the evening.

226. The Sampling of Effluents.—Take a clean bottle of about a pint capacity, rinse it out well with clean water, and proceed to take the sample Half fill the bottle with the effluent, and pour out again Now fill the bottle quite full, leaving just sufficient room for the stopper The bottle should be fitted with a ground glass stopper, *a cork should not be used* This sample should be put aside, the bottle being tightly stoppered, and at the right intervals the second, third, and fourth samples should be drawn in like manner. When the four samples have been collected, pour the contents of the bottles into a larger vessel, and mix thoroughly. If much suspended matter or sediment is present in the separate samples, the necessity for this thorough mixing is the more urgent. Now fill two bottles with the final sample One of these should be sealed, marked with the date and hour of sampling, and kept for reference The other should be tested as follows:—

227. Suspended Matter (Suspended Solids).—The suspended matter is estimated as described in paragraph 208 Shake the sample well before measuring off the quantity to be tested. This is a precaution which must be very carefully observed, otherwise the result may be altogether misleading Estimate the "loss on ignition," after weighing off (see paragraph 210). The residue after ignition = mineral; loss = volatile.

228. Total Solids.—The method for the estimation of the total solids will be found in paragraph 207. In measuring off the two lots of 70 c.c. for the purpose of this test, the precaution with regard to the shaking of the sample must also be carefully

[illegible]

observed Estimate the loss on ignition, after weighing off (see paragraph 210) Residue after ignition = mineral, loss = volatile

229. Solids in Solution (Dissolved Solids).—About 250 c c. of the shaken sample are filtered as in the case of ordinary water (see paragraph 209), and two lots of 70 c.c are evaporated to dryness as there described Estimate the loss of ignition (see paragraph 210) Residue after ignition = mineral; loss = volatile

In the execution of each of the foregoing three tests the method of operation is precisely the same as in the case of ordinary waters Before testing, the samples must be thoroughly agitated, but in other respects the tests are identical.

230. Organic Matter.—For trade purposes, the "Loss on Ignition" may be taken as a measure of the organic matter contained in the effluent (see paragraph 210) Carry out the test in each case in exactly the same way as indicated therein.

231. Total Hardness.—See paragraph 211. The total hardness in an effluent is estimated in the same manner as in an ordinary water The hardness is much higher, however, and it will frequently be found necessary to dilute the sample with distilled water, when the hardness is above 17°.

232. Permanent Hardness.—The test is the same as for an ordinary sample of water (See paragraph 212)

233. Temporary Hardness.—This is the difference between the total and permanent hardness (See paragraph 213.)

234. Alkalinity.—Measure off 350 c c. of the water, add 5 c c. of normal sulphuric acid (paragraph 48), and evaporate to 100 c c. Cool, and titrate with normal caustic alkali (see paragraph 49), using methyl orange as indicator. Deduct the c.c. of alkali used from the 5 c c. acid, and multiply this figure by 200. This gives the number of grains per gallon of alkalinity in terms of sulphuric acid neutralised

235. Absorbed Oxygen in Waters.—The following is the test used by the Mersey and Irwell Joint Rivers Committee.—

Solutions Required—(1) Dilute sulphuric acid; add one volume pure sulphuric acid to three volumes distilled water, and colour with a little solution of potassium permanganate. (2) Solution of potassium permanganate: weigh off 0·395 gm of permanganate, and dissolve in one litre of distilled water (3) Solution of sodium hyposulphite. weigh off 4 gm. of pure sodium hyposulphite, and dissolve in one litre of distilled water (4) Solution of potassium iodide: weigh off 10 gm. pure potassium iodide, and dissolve in 100 c c. of distilled water. (5) Starch solution this is prepared by adding nearly boiling distilled water to a little starch; cool, settle, and use the clear liquid only

The Test.—Four hours Measure off 70 c.c. of the water into a stoppered white-glass bottle holding about 8 oz; add 10 c.c sulphuric acid solution, and 50 c c. permanganate solution. Place in a dark cupboard, and leave for four hours at about 60° F. If the solution fades in colour very much, add a second 50 c c permanganate At the end of four hours add a few c c. potassium iodide solution, and then titrate with hyposulphite solution until the colour is almost gone; add a few drops starch solution, and titrate until the colour is discharged. *(Three minutes' test)* · Run the 70 c c. sample into the bottle, add 50 c.c. permanganate and 10 c.c. sulphuric acid, allow to stand three minutes in cupboard at about 60° F, and titrate as above.

Blank test—A blank test is now made by taking 70 c c. distilled water, 10 c.c. acid, and 50 c.c permanganate, and titrating as in the case of the effluent.

Calculation—

$$\frac{\text{c.c. hypo used} \times 5}{\text{c.c. hypo used in blank test}} = \text{grains per gallon of absorbed oxygen.}$$

236. Testing against River Inspectors.—The method in which the analyses of works effluents are submitted by the river authorities is indicated by the table here given (paragraph 237). From this some idea may be gathered as to the requirements to be observed in order to reach the standard of a "good effluent."

The great difficulty in dealing with Rivers Committees, Conservancy Boards, and other bodies controlling the rivers, is that it is next to impossible to get them to fix a standard of purity to which manufacturers may strive to attain Their Inspectors draw samples regularly of the effluents turned into the rivers by manufacturers, and on the analysis of these samples they intimate to him that he is not fulfilling their requirements; but if a manufacturer, in despair, should demand what constitutes a good effluent on their standard, they will take very good care that definite information is not communicated. Under these circumstances, it is by no means easy for a manufacturer, whose effluent is at times liable to be bad, to avoid prosecution, but it *is* possible for him to take considerable precautions to prevent such an undesirable state of things. The figures in paragraph 237 give some idea of what the rivers authorities regard as a good effluent, therefore, if a manufacturer can so control his waste liquors that the suspended solids, the total solids, the dissolved solids, and the absorbed oxygen are not greater, but rather less, than the figures given under the head of a good effluent, he need have little fear of being prosecuted for rivers pollution. On the other hand, if he is detected in the act of turning into the river an effluent which is admittedly bad, it would considerably strengthen his position if he could produce a record which would prove that, so far from being the rule, the bad sample was an exception, and that his average effluent was well below the limits in polluting matter In the face of such a record, a manufacturer who had been unfortunate in a single instance would be assured of much more considerate treatment at the hands of the authorities than would be the case when, as generally happens, the only evidence is the incriminating certificate of the chemist to the Rivers Board An even more probable conclusion, however, is that a manufacturer who maintained the control of his effluent as here indicated would never find himself in trouble, as the greatest safeguard against pollution is the knowledge that such pollution is probable, when the necessary steps may at once be taken to prevent its occurrence, without waiting for an inspector to supply the information

237. Official Report on Manufacturers' Effluents.—

(Results expressed in parts per 100,000) :—

NATURE OF SAMPLE.	DISSOLVED SOLIDS			SUSPENDED SOLIDS.			
	Mineral	V'latile.	Total.	Mineral	Volatile.	Total	
Tank effluent	19	*21*	40		·2	·2	Good.
Filter effluent ..	41	*21*	62	·0	0 2	·2	
Filter effluent	52	*26*	78	·0	1 0	1 0	
Filter effluent ..	100	*43*	143	·2	·8	1 0	
Filter effluent .. .	41	*19*	60	·0	1·0	1 0	
Effluent from lodge	56	*32*	88	·6	1·0	1 6	
Effluent from lodge	41	*22*	63	·4	1 2	1 6	
Filter effluent	32	*28*	60	·6	1 4	2·0	
Tank effluent	41	*24*	65	2	2·8	3 0	
Tank effluent	74	*48*	122	·6	2 8	3 4	
Tank effluent	44	*18*	62	1·4	2·4	3 8	
Tank effluent	39	*26*	65	·8	3 2	4·0	
Tank effluent	48	*25*	73	1·0	3·4	4 4	
Tank effluent .	37	*34*	71	1·4	4·0	5·4	
Tank effluent .. .	199	*132*	331	4 0	12 0	16 0	Bad.
Tank effluent	179	*156*	335	3 7	13 6	17 3	
Tank effluent	214	*170*	384	12 0	12 0	24 4	
Filter effluent	995	465	1460	.			Objectionable
Filter effluent	4060	960	5020	..		..	

Note.—The above results are expressed in parts of 100,000; parts per 100,000 × 0·7 = grains per gallon The tests in this chapter are for grains per gallon; if the results are to be expressed as parts per 100,000, then 100 c.c. of sample are taken in each case instead of 70 c.c.

Ammonia in Effluents.—The rivers authorities do not usually estimate the free and albuminoid ammonia in manufacturers' effluents. The table given in paragraph 237 shows the estimations carried out by the controlling chemist, so that if the works record shows the results under the same headings as those given, this will be sufficient for general purposes. With regard to ammonia in effluents, a study of results of the [illegible] the following conclusions:—

Albuminoid ammonia [illegible]

Absorbed Oxygen.—This is not shown in the returns of manufacturers' effluents, but the method is given in paragraph 235 Absorbed oxygen appears to be estimated when the albuminoid ammonia exceeds 0 150 parts per 100,000.

237a. Purification of Effluents.—Where the impurities in trade effluents consist chiefly of suspended matters, the greater part of these may be removed by subsidence in suitable tanks, and subsequent filtration The construction of the filters generally in use for this purpose is well known, and consists essentially of several layers of filtering material of varying size, the top layer being generally cinders

The question of the purification of effluents by precipitation with chemicals and subsequent filtration should be systematically studied The best method of procedure is as follows: First estimate the solid matters in solution as described in this chapter. Then add varying proportions of the precipitants—which are generally lime, copperas, or alum—until the best results are obtained, as indicated by careful tests of the treated effluent, after subsidence and filtration Having obtained reliable figures from such experiments, a careful study of these will show which precipitant, and in what proportion, will give the best results. In this connection, the cost of each precipitant per 1,000 gallons of effluent treated must be considered, in comparison with the percentage of purification effected by its use. Other things being equal, the cheapest reagent will, of course, be selected This having been done, the quantity to be employed per per 1,000 gallons should be carefully calculated, and no great excess should be used The tests given in this chapter will, if properly carried out, supply all the data requisite for the efficient control of trade effluents

CHAPTER X.

Miscellaneous Testing, Tables, &c.

TEXTILE SUBSTANCES.

The complete testing of textile substances, and the estimation of the exact conditions in which they are commercially handled, is beyond the scope of the present work. It is here proposed to deal only with the testing of goods for moisture, and fat, grease, or oil These are questions which occasionally come up for decision, and the following tests may be useful —

238. Moisture.—This is estimated by drying at a uniform temperature (100° C.) until the weight is constant. The sample should be a representative one, whether of yarn, weft, or cloth, and whether in cop, hank, or piece The sample for testing, representing the whole of the parcel in question, is dried in the water oven at 100° C., cooled in a tightly-closed vessel, and weighed accurately when cool The percentage of moisture is then calculated from the loss on drying

239. Fat, Oil, or Grease.—Weigh off a quantity of the sample (from 10 to 20 gm), and pack loosely into the tube of a Soxhlet's extractor (see paragraph 150) Clean and weigh accurately a small flask holding about 200 c c , with round bottom Fit up the apparatus as shown in Fig. 34; pour about 100 c.c. of benzol into the flask, and commence gentle distillation (using a sand bath for heating) At first the solvent will run through coloured; as the test proceeds it will come through colourless, as in the case of the extraction of sulphur in spent oxide (paragraph 150). Continue the distillation for some time after this takes place, then cool down; remove the flask, distil off the benzol (which should be kept in a special bottle, and labelled " benzol for extraction of fats "); place the flask in the water oven (first removing the vapour by exposing to the air for a time), dry and weigh until the weight is constant. The weighings must be made carefully, as the residue in many cases is small. The difference in weight of the flask gives the fat, oil, or grease contained in the weight of the sample extracted; if 10 gm. are taken, multiply by 10, and the result is the percentage contained in the sample.

BOG ORE FOR GAS PURIFICATION

Bog ore, which is chiefly used for gas purification, is generally tested for moisture and hydrated oxide of iron.

240. Moisture.—Crush and mix well; if the sample is a large one, reduce by quartering. Weigh off 5 gm. into a porcelain or platinum basin, and dry in the water oven at 100° C. until the weight is constant. The loss, multiplied by 20, gives the percentage of moisture.

241. Hydrated Oxide of Iron.—Make up a solution of hydrochloric acid of 6° Tw. strength. Place 5 gm. of sample into a beaker with 200 c.c. of the hydrochloric acid; stand on the water bath, and digest for some time. Filter into a 500 c.c. flask, make up to the mark when cold, and mix. Measure off 50 c.c. into a beaker, add a little potassium chlorate (crystals), boil well, and add caustic soda solution until the liquid is strongly alkaline. Filter, wash free from alkali, spread out the filter paper on a clock glass over the water bath, and dry (finishing off in the water oven). Place the filter paper with precipitate in a large porcelain crucible or a platinum dish, and ignite over a strong Bunsen burner, or in the muffle furnace, cool under the desiccator, and weigh the ferric oxide. Divide the weight of ignited oxide by the dry weight of 5 gm. sample; multiply this figure by 1338, and the result is the percentage of hydrated oxide of iron.

HYDROCHLORIC ACID

241a. Specific Gravity.—Take this carefully by means of the Westphal balance (paragraph 18).

Hydrochloric Acid.—Pipette off carefully 10 c.c. with an accurate pipette, dilute to 200 c.c., pipette off 10 c.c. of this, add pure sodium carbonate until faintly alkaline, colour with potassium chromate solution, and titrate with decinormal silver nitrate (see paragraph 112). Deduct 0·2 c.c. from silver solution consumed, multiply by 73, and divide by the specific gravity of the acid. The result is percentage of hydrochloric acid.

Sulphuric Acid.—This is estimated by saturation with pure carbonate of soda (free from sulphate) and precipitating with barium chloride (see paragraph 88), 1 part of barium sulphate = 0 34335 SO_3.

Iron.—See paragraph 100

NITRIC ACID

241b. Specific Gravity.—This should be carefully taken with the Westphal balance

Impurities.—*Fixed residue* Evaporate to dryness a measured quantity of the acid, and weigh the ignited residue *Sulphuric acid* · See paragraph 241a (sulphuric acid). *Chlorine* The acid is saturated with pure carbonate of soda until neutral or faintly alkaline, coloured with bichromate of potash solution, and titrated with silver nitrate (see paragraph 97). *Iron* See paragraph 100

BEESWAX

241c. For the complete analysis of beeswax, Allen's "Commercial Organic Analysis," Vol II (J. & A. Churchill) should be consulted. The following tests serve for the detection of the adulterants usually found in the commercial article .—

Water.—Weigh off 10 gm of sample in a dish, melt, and keep the molten wax at a temperature of 110° C until no more globules of water are observed. Cool and weigh. The loss × 10 = per cent. water.

Mineral Matter.—Weigh off 5 gm , and ignite in a platinum dish or large crucible. Burn off completely, cool, and weigh. Residue × 20 = per cent mineral matter

Sulphur.—Boil with a weak solution of soda, and add acetate of lead solution when cool If sulphur is present, a black or brown precipitate will result.

Starch and Flour.—Treat 10 gm of sample with warm turpentine, filter through a weighed paper, dry, and weigh. Residue × 10 = percentage.

Japan Wax, Tallow, &c.—Boil 1 gm. of sample with 1½ gm. of borax and 20 c.c. water If pure, the liquid remains practically clear. If the above impurities are present, the liquid will be milky or gelatinous.

CHIMNEY GASES.

241d. Hydrochloric Acid.—Two bottles are partly filled with distilled water, and one cubic foot of the gas is drawn through. To the liquid, pure bicarbonate of soda is added until faintly alkaline, then coloured with pure bichromate of potash solution, and a solution of silver nitrate containing 3·02 gm., in a litre of distilled water, is added (see paragraph 112). Each c.c. = 0·01 gm. hydrochloric acid per cubic foot.

241e. Total Acidity.—According to the Alkali Act, the "total acidity" of the exit gases from vitriol plants, etc., must not exceed 4 grains of SO_3 per cubic foot. The total acidity may be estimated as follows. By means of a suitable aspirator the air is drawn through four absorbing bottles; the first three contain 100 c.c. standard caustic soda solution, and the fourth distilled water, at least 3 in. of liquid being contained in each bottle, and the inlet tubes being not larger than 1/50th of an inch in diameter. The contents of the four bottles are united, with as little dilution as possible, well mixed, and divided into three equal portions. One-third is titrated with standard sulphuric acid, using phenolphthalein as indicator. The calculation is as follows. If V = volume of air used (reduced to 0° C. and 760 m.m. pressure) and x = c.c. standard acid used in titration, then

$$\frac{1{\cdot}852 \times (100 - x)}{V} =$$

total acidity in grains per cubic foot. If chimney testing is to be undertaken, the reader should first consult Lunge and Hurter's "Alkali Makers' Pocket Book" on the subject.

BOILER FLUIDS.

242. The principal test to be applied to boiler fluids is the estimation of the alkalinity. As a basis for comparison this is the most convenient test. Measure off 25 c.c. of the sample, and dilute to 500 c.c. Pipette off 50 c.c. into a flask, dilute rather freely with water to reduce the colour, add a few drops of methyl orange, and titrate with normal acid (see paragraph 106).

SOAP.

243. Water.—Clean and weigh a platinum or porcelain dish. Remove the outer crust from the sample of soap, and from the inner portion scrape off 10 gm. into the dish. Dry in the water oven for about three hours, cool under the desiccator, weigh, return to the oven for an hour, cool, and weigh again. Repeat this until the weight is constant. The loss, multiplied by 10, gives the percentage of water

244. Unsaponified Fat (Free Fat).—Extract the dried sample, after the estimation of the water, in a Soxhlet's apparatus (see paragraph 239) Wrap the soap in a piece of filter paper, or a cartridge of filter paper; weigh the cleaned and dried flask as in sulphur extraction (paragraph 150), and exhaust with petroleum ether for about an hour and a half The solvent is distilled off as in paragraph 239, and the residue weighed This, multiplied by 10, gives the percentage of unsaponified fat

245. Free Alkali.—Weigh off 10 gm of the shredded sample, and boil in about 200 c c. of water, add a little phenolphthalein (paragraph 64), and titrate with standard sulphuric acid (paragraph 48) Phenolphthalein gives a red colour in presence of free alkali, and this colour disappears on neutralization with acid The c.c. of sulphuric acid used, multiplied by 10, and then by 0 04, gives the percentage of caustic soda (Hurst, in his work on "Soaps," states that 10 gm of a good soap should not consume more than 0 75 to 1 c c of standard acid)

THE DETECTION OF ARSENIC

It is not proposed to describe here the methods to be used in the quantitative estimation of minute proportions of arsenic. This task would, no doubt, generally speaking, be somewhat out of the reach of the reader of this volume, which, moreover, does not contain an exposition of exact analysis where such minute quantities are involved as in the case of arsenic in many products.

The recent epidemic of arsenical poisoning from contaminated beer has resulted in the discovery that arsenic is present in many raw materials, where such an impurity had previously never been suspected The consequence is, that users of materials in which arsenic may possibly exist as an impurity, are becoming aware of the necessity for testing to ensure the absence of this undesirable substance. It is proposed to embody in this chapter such instructions as will serve to detect the presence of arsenic, if it is desired to estimate the exact quantity present, the reader is referred to the booklet issued by the Society of Chemical Industry, entitled "The Detection and Determination of Arsenic," which may be obtained, price 1s., from Messrs Eyre and Spottiswoode, East Harding Street, London, E.C., and also to the recently published work on "Arsenic," by Professor Wanklyn. The details which follow will answer the purpose of the investigator who wishes to prove the presence or absence of arsenic in his raw materials and manufactured products; beyond this, in the space here available, it is impossible to go.

246. Marsh's Test for Arsenic.—Hydrogen is generated in a small apparatus—a Woulff's bottle for preference. A few pieces of zinc rod (free from arsenic) are introduced, together with dilute sulphuric acid, also arsenic-free. To the delivery tube of the hydrogen apparatus, a piece of glass tubing, drawn out to a fine jet, is attached by a rubber joint. The evolution of gas is started, and after a time a light is applied to the jet. This must not be done too soon, or an explosion will result. The gas may be tested by filling a test tube, and holding the mouth to a Bunsen burner; if the gas burns quietly, the jet may be ignited. A cold white porcelain dish or tile is held in the flame; if no deposit forms, the apparatus is clean, and ready for the test.

A *little* of the solution to be tested is poured down the funnel of the hydrogen apparatus, and the cold white porcelain is again held in the flame; if arsenic is present, a metallic film will be deposited of a brownish-black colour. The flame must not be allowed to play on the film for more than a moment, or the film will be volatilized. If a part of the jet-tube is heated, a brilliant mirror of arsenic is produced in the tube. The film produced on the porcelain may be proved to be arsenic by its solubility in a solution of hypochlorite of soda (bleaching powder). Antimony produces a metallic mirror, but the stain does not dissolve in hypochlorite solution.

247. Reinsch's Test for Arsenic.—If a solution mixed with hydrochloric acid is heated with a clean piece of copper foil or wire, a grey film of copper arsenide is formed on the copper, even in very dilute solutions. If the deposit is dried, the arsenic is volatilized and oxidised, giving a white crystalline precipitate of arsenious oxide.

247a. Modified Reinsch's Test for Arsenic in Beer.—The Commission of Experts appointed by the Manchester

Brewers' Central Association, in 1901, recommended the following method of performing the Reinsch test:—

The Commission recommend that the Reinsch test should be used in preference to all others at present known, because their investigations have satisfied them that it is the best and most reliable test for arsenic in beer. The mode of performing it is as follows: Take 200 c.c. of the beer in a porcelain evaporating dish. Raise the liquid to the boiling point, and then add 30 c.c. of pure concentrated hydrochloric acid. Insert a piece of pure bright copper foil, about a quarter of an inch by half an inch in size, and keep the solution gently boiling for forty-five minutes. If, at the end of that time, the copper remains bright and red, the beer is free from arsenic.

If a deposit is obtained on the copper, the foil is to be washed successively with water, alcohol, and ether (care being taken that these are pure), dried at a temperature not exceeding 100° C., and subjected to slow sublimation in a thin reduction tube of small section, and not less than two inches long, the upper portion of which should be warmed before the sublimation begins. For the purpose of sublimation a small spirit lamp flame should be used. If any sublimate is obtained, it must be examined under a magnifying power of about 200 diameters. Any sublimate which does not show well-defined octahedral or tetrahedral crystals is not to be considered arsenical.

It must be borne in mind that the blackening of the copper or a deposit thereon from the preliminary operation does not demonstrate the presence of arsenic in beer. Abundant blackening and deposit may be obtained from the purest beer.

248. Arsenic in Coal and Coke.—The following test, for the detection of minute quantities of arsenic in coal and coke, was described at length in a paper read before the Nottingham Section of the Society of Chemical Industry, on April 24th, 1901, by Messrs. L. Archbutt and P. G. Jackson:—

A fair average sample of the coke, obtained from a large bulk by crushing and quartering in the usual manner, is reduced to coarse powder in a grinding mill, and rather more than 50 gm. of this coarse powder are further ground in a mortar and passed through a sieve, having 90 meshes in one linear inch, then dried at 100° C. Of the dry coke, 50 gm. are placed in a 16 oz. plain retort, and heated for at least two hours with 100 c.c. of pure nitric acid (1·42 specific gravity), the neck of the retort being inclined upwards, so as to condense and return the acid which evaporates. A small flame is used, large enough to maintain gentle ebullition. Copious red fumes are evolved.

The contents of the retort are transferred to a beaker, diluted with hot distilled water to about 400 c.c., and, after settling, filtered, and the coke washed by suction on a toughened filter, which is easily done. The clear filtrate is evaporated in a porcelain basin to complete dryness on a water bath, the residue is mixed with 25 c.c. of pure concentrated sulphuric acid, and further heated, with frequent stirring, until the ferric nitrate is decomposed, and a smooth cream containing ferric sulphate and free sulphuric acid obtained. Heating is then continued over a sand bath until sulphuric acid fumes come off. The residue, when cold, is diluted with enough water to dissolve it on warming, and again evaporated until sulphuric acid fumes appear. This is repeated until, on diluting with water, no smell of nitric acid is observed—the second evaporation being usually sufficient. To make quite sure of expelling every trace of nitric acid, solution and evaporation are repeated once more.

The residue is next dissolved in 50 c.c. of water, and transferred to a 16 oz. distillation flask connected to a Thorpe's revenue condenser, having an adapter attached to the end, which dips below the surface of about 20 c.c. of water contained in the receiving flask. The distillation flask is closed by a rubber bung, through which is passed a thermometer having its bulb within ¼ inch of the bottom of the flask. Then 23 gm. of purified chloride of sodium and 2 gm. of ferrous sulphate are added to the liquid in the flask, which should be quite cold, and not more than 100 c.c. in volume. Distillation commences at about 110° C., and is continued until the temperature reaches 125° C., at which point the whole of the arsenic will be found in the distillate.

The authors did not succeed in titrating these minute traces of arsenic in the distillate direct, after neutralization with ammonia, the end of the reaction being too indistinct. They therefore precipitated the arsenic as sulphide, by adding to the acid distillate some pure sulphide of zinc, and agitating for a few minutes until the precipitate coagulated. The delicacy of this re-action is said to be remarkable—as little as 0·1 milligramme of As_4O_6 in 100 c.c. being recognisable in daylight by the yellow colour produced on adding the sulphide of zinc, and after agitating for a few minutes, even so small a precipitate coagulates and can be filtered off.

249. British Pharmacopœia Tests for Arsenic (B.P., 1898).—Sulphuretted hydrogen gives, in solutions containing hydrochloric acid, a yellow precipitate, soluble in solution of caustic potash, potassium carbonate, ammonium hydrosulphide, and potassium hydrogen sulphite, and in solution of B.P. ammonium carbonate, but reprecipitated on addition of hydrochloric acid. The precipitate is insoluble in the strongest hydrochloric acid.

Nascent hydrogen, generated by the action of zinc and diluted sulphuric acid, converts arsenium compounds into arseniuretted hydrogen. A cold porcelain tile, held in the flame of this gas, acquires a dark metallic deposit, which is readily dissolved by solution of chlorinated soda. The gas, when passed into excess of solution of silver nitrate, causes a black precipitate of silver, and the cautious addition of ammonia solution to the supernatant liquid causes a yellow precipitate.

Stannous chloride, dissolved in a large excess of hydrochloric acid, gives, on boiling with a solution containing arsenic, a brownish-black precipitate.

Bright copper foil precipitates arsenic from solutions acidified by hydrochloric acid, and the arsenic may be volatilized by heat in an open tube, when it condenses, at some distance from the copper, as a white sublimate of characteristic octahedral crystals.

Arsenites.—Solutions of arsenites give a yellow precipitate with a solution of silver ammonio nitrate.

Arsenates.—Solutions of arsenates give a reddish-chocolate precipitate with solution of silver ammonio nitrate.

250. Arsenic in Glycerine (British Pharmacopœia Test, 1898).—Two c.c. of the glycerine are diluted with 5 c.c. of a mixture of 1 part of pure hydrochloric acid and 7 parts of water; 1 gm. of pure zinc is added, and the whole placed in a long test tube, the mouth of which is covered by a piece of filter paper moistened with a drop or two of mercuric chloride solution, and dried. The paper should not show a yellow stain on the paper even after 15 minutes (limit of arsenic).

QUALITATIVE ANALYSIS

The reader is recommended to secure a good text-book of qualitative analysis—there are several available, such as Newth's "Manual of Chemical Analysis Qualitative and Quantitative," (Longmans & Co.); or Clowes and Coleman's "Qualitative Chemical Analysis" (J. & A Churchill) The instructions for general manipulation should be studied as therein indicated, as, without such preparation, it will be very difficult for the novice to understand and execute the tests involved, particularly where the separation of a number of metals is to be undertaken. A set of tables, illustrating the method of separation recommended by Fresenius, will be found in this chapter, and will be useful as a guide to general analytical work of a qualitative nature.

Reactions of the Metals

251. **Silver.**—*Dry* (1) Mixed with sodium carbonate, and heated with charcoal under the blow pipe, compounds of silver give brilliant metallic globules

Wet (2) Hydrochloric acid gives a white curdy precipitate, insoluble in nitric acid, soluble in ammonia

252. **Monad Mercury.**—*Dry* (1) Mercurous compounds volatilize when heated in a glass ignition tube. (2) Mixed with dry carbonate of soda and heated in an ignition tube, covered with a little dry carbonate of soda, the mercury sublimes, forming a grey ring.

Wet (3) Hydrochloric acid gives a white precipitate, insoluble in cold hydrochloric acid and cold nitric acid, soluble on prolonged boiling in these acids (4) Sulphuretted hydrogen gives a black precipitate, insoluble in dilute acids and sulphide of ammonia, but soluble in sulphide of soda in presence of caustic soda, and readily soluble in *aqua regia* (5) Ammonium sulphide gives same precipitate as (4) (6) Tin chloride gives a grey precipitate.

253. **Lead.**—*Dry* (1) Mixed with carbonate of soda, and heated on charcoal under blow pipe, malleable metallic beads are obtained.

Wet· (2) Hydrochloric acid gives a crystalline precipitate, soluble in water. (3) Sulphuretted hydrogen gives a black precipitate, insoluble in cold dilute acids, soluble in hot dilute nitric acid. (4) Ammonium sulphide gives a black precipitate. (5)

Sulphuric acid gives a white precipitate (6) Potassium bichromate gives a yellow precipitate (7) Potassium iodide gives a yellow precipitate.

254. Dyad Mercury.—*Wet.* Sulphuretted hydrogen, in small quantities, gives, on shaking, a white precipitate, a larger quantity causes precipitate to turn yellow, and excess gives a black.

255. Bismuth.—*Dry* Compounds of bismuth give brittle metallic beads when heated with carbonate of soda on charcoal

Wet (1) Sulphuretted hydrogen gives a black precipitate, insoluble in dilute acids and alkaline sulphides, readily dissolved by boiling nitric acid (2) Water forms a white precipitate.

256. Copper. *Dry* In the Bunsen flame copper gives an intensely green colour, hydrochloric acid intensifies this colouration. (2) The borax bead gives a green colour when hot, and blue when cold.

Wet (3) Sulphuretted hydrogen gives a brownish-black precipitate, insoluble in dilute acids and caustic alkalies, readily soluble in boiling nitric acid, and *slightly* soluble in sulphide of ammonia; completely soluble in potassium cyanide (4) Ammonia in excess gives a deep azure colour.

257. Cadmium.—*Wet* Sulphuretted hydrogen gives a bright yellow precipitate, insoluble in dilute acids and alkalies, easily soluble in boiling nitric, hydrochloric, and sulphuric acid

258. Tin.—*Dry:* (1) Fused on charcoal with carbonate of soda and cyanide of potassium, malleable beads of metallic tin are formed.

Wet: (2) Mercuric chloride in excess gives a grey or white precipitate. (See Group II, Separations.)

259. Antimony.—*Dry reactions:* (1) Brittle beads of metallic antimony are produced by heating with carbonate of soda on charcoal, which burn and sink in the charcoal, giving a white film.

Wet reactions: (2) Sulphuretted hydrogen in acid solution gives an orange-red precipitate, soluble in caustic potash and alkaline sulphides, insoluble in dilute acids, soluble in boiling strong hydrochloric acid. (3) Zinc gives a black powder. (See Group II., Separations.) (4) Solutions of antimony, by Marsh's test (paragraph 246) give mirrors *insoluble* in chloride of soda solution (arsenic is *soluble*).

260. Arsenic.—See paragraphs 246 and 247.

261. Selenium.—The test of the United States Pharmacopœia for selenium in sulphur is to boil equal parts of the sulphur and potassium cyanide in water, to filter, and add excess of hydrochloric acid to the filtrate If selenium is present, a reddish colouration results, and it is said that the delicacy of this test is such as to enable the detection of 1/500th of a grain of selenium

262. Nickel.—*Dry* Heated in a borax bead, a reddish-brown bead is obtained

Wet Ammonium sulphide gives a black precipitate, not completely soluble in excess of sulphide, and so giving a dark filtrate

263. Cobalt.—*Dry* In borax bead a bright blue bead is obtained This test is very delicate

Wet Ammonium sulphide and chloride give a black precipitate, insoluble in dilute hydrochloric acid and alkalies, and soluble in hot *aqua regia*

264. Iron.—*Dry* In borax bead, in the inner flame, a bead is obtained which is bottle-green when cold.

Wet Potassium ferrocyanide gives a blue precipitate with ferric salts Add a little nitric acid to oxidise the iron before testing Potassium sulphocyanide gives a red colour with ferric solutions

265. Manganese.—*Dry* In the outer flame, the borax bead is coloured red when cold A carbonate of soda bead, touched with a finely-divided manganese compound, held in the flame, then dipped into powdered potassium nitrate, and heated again, gives a green bead, bluish on cooling This test is a very delicate one

266. Zinc.—*Wet.* Ammonium sulphide in presence of ammonium chloride gives a white precipitate, insoluble in ammonium sulphide, potash, and ammonia, soluble in hydrochloric, nitric, and dilute sulphuric acid Sulphuretted hydrogen gives the same precipitate Carbonate of soda gives a white precipitate, insoluble in excess of soda. Potassium ferrocyanide gives a white, slimy precipitate, insoluble in hydrochloric acid, partly soluble in excess of the precipitant. Ferricyanide of potassium gives a brownish-orange coloured precipitate, soluble in ammonia and hydrochloric acid.

267. Chromium.—*Dry* Borax bead is coloured emerald when cold. Chromium compounds, when fused with chlorate of potash and sodium carbonate on porcelain or platinum, give a yellow mass.

Wet: Chloride of soda solution gives a yellow liquid on boiling; lead acetate gives a yellow precipitate

268. Aluminium.—*Wet:* Caustic potash and ammonia gives a white precipitate, which, on platinum wire in Bunsen flame, glows brightly.

269. Barium.—*Dry:* Barium compounds give a yellowish-green flame when heated on platinum wire

Wet: Sulphuric acid and sulphate of calcium both give a heavy white precipitate, even in dilute solutions, insoluble in alkalies, and almost so in dilute acids; perfectly soluble in boiling nitric and hydrochloric acids. Bichromate of potash gives a light yellow precipitate, soluble in nitric and hydrochloric acids

270. Strontium.—*Dry:* Moistened with strong hydrochloric acid and heated in the Bunsen flame, strontium compounds give an intensely red colour.

Wet: Ammonium carbonate gives a white precipitate. Sulphuric acid and calcium sulphate give a white precipitate after standing, soluble to a certain extent in hydrochloric and nitric acids. Bichromate of potash precipitates a light yellow crystalline precipitate on long standing.

271. Calcium.—*Dry:* Calcium compounds give a yellowish-red flame.

Wet: Ammonium carbonate gives a white precipitate; oxalate of ammonia, white precipitate; phosphate of soda, white precipitate; sulphuric acid, white precipitate. Test these precipitates in flame.

272. Magnesium.—*Dry:* Magnesium compounds glow brightly in flame.

Wet: Phosphate of soda gives white precipitate Ammonia alone gives gelatinous precipitate, soluble in ammonium chloride Phospate of soda gives a white precipitate—crystalline, and most marked on standing.

273. Potassium.—*Dry:* Violet flame. *Wet:* See Group V, Separations.

274. **Sodium.**—*Dry*. Intensely yellow flame.

275. **Ammonia.**—Heated with quicklime or caustic soda gives off distinct ammonia smell, and platinic chloride gives a heavy yellow precipitate in strong acid and neutral solution Ammonia gas turns a red litmus paper a blue colour.

REACTIONS OF THE ACIDS

276. **Sulphate.** — A portion of the substance is boiled with hydrochloric acid, filtered, and tested with barium chloride A white precipitate shows the presence of a sulphate

277. **Chloride.**—A portion of the substance is warmed with nitric acid, filtered, and silver nitrate solution added A white precipitate, easily soluble in warm ammonia, shows the presence of a chloride

278. **Nitrate.**—The clear solution of the substance is mixed with its own volume of strong sulphuric acid, and the liquid cooled. Freshly-made cold solution of ferrous sulphate is poured as a layer upon the surface of the liquid; a brown ring at the point of contact shows the presence of a nitrate

279. **Cyanide.**—This, if present, may be detected by the smell of bitter almonds which is given off by the substance on heating with sulphuric acid.

280. **Phosphate.**—Boil some of the original substance with dilute nitric acid, and add a little of the clear solution to some ammonium molybdate. Shake and mix the liquid well, and if no precipitate forms warm very gently A yellow precipitate shows the presence of a phosphate

281. **Borate.**—Warm some of the substance with a little dilute hydrochloric acid, dip into the solution a strip of turmeric paper, and dry the paper at a gentle heat The change of colour of the turmeric to reddish-brown, becoming blue-black when moistened with ammonia, shows the presence of a borate.

282. **Fluoride.**—Place the powdered substance in a lead cup or platinum crucible with strong sulphuric acid Coat a watch glass with a film of paraffin wax, and scratch lines through the film with the point of a penknife Place the watch glass as a

cover upon the metal vessel, and gently warm Remove the wax If the lines are etched upon the glass, the presence of a fluoride is shown.

283. Sulphide.—This is easily detected by the smell on heating with strong hydrochloric acid A strip of filter paper, dipped in lead acetate, is turned black

TABLES FOR QUALITATIVE ANALYSIS

284. The following tables show the methods to be followed in the separation of the metals It is assumed that the solution to be dealt with contains *all* the common metals The detection of these, and the means by which they may be separated, are indicated —

Group I.—Add hydrochloric acid; silver, monad mercury, and lead are precipitated

Group II.—Filter, pass sulphuretted hydrogen through the filtrate, dyad mercury, bismuth, copper, cadmium, tin, antimony, and arsenic are precipitated.

Group III—Filter, add ammonium chloride solution, ammonium hydrate, and ammonium sulphide; nickel, cobalt, iron, manganese, zinc, chromium, and aluminium are precipitated.

Group IV.—Filter, add ammonium carbonate solution, barium, strontium, and calcium are precipitated

Group V.—The filtrate contains magnesium, potassium, sodium, and ammonium.

285. Group I.—The Precipitate from Hydrochloric Acid.—Wash the precipitate on filter with hot water.

FILTRATE.

Add sulphuric acid : White precipitate = *lead.*

RESIDUE.

Wash on filter with hot water

Filtrate.

Add excess of ammonia · White precipitate = *silver.*

Residue.

If black = *mercury*

286. Group II.—Precipitate from Sulphuretted Hydrogen.—Warm the precipitate with ammonium sulphide solution, and filter :—

RESIDUE FROM AMMONIUM SULPHIDE.

Wash, boil in strong nitric acid, and filter :—

Residue

Divide in two parts :

(1) Dissolve in hydrochloric acid with chlorate of potash, and add tin chloride solution : Grey or white precipitate = *mercury.*

(2) Fuse with potassium cyanide and sodium carbonate, if metallic globules, wash them and treat with nitric acid, and filter.

Precipitate.

Wash, boil with strong hydrochloric acid, pour off acid, add water, and then pass sulphuretted hydrogen. Yellow precipitate = *tin*

Filtrate

Add sulphuric acid White precipitate = *lead.*

Filtrate

Add dilute sulphuric acid, evaporate on water bath till nitric acid is driven off, take up with water, and filter Residue = *lead.* To the *filtrate*, add excess of ammonia, warm, and filter.

Precipitate.

Wash, dissolve in a watch glass in a *little* hydrochloric acid, add water. Milkiness = *bismuth*

Filtrate

(If blue, *copper* is present), pass sulphuretted hydrogen gas through, wash precipitate, boil with dilute sulphuric acid, filter off copper sulphide and pass sulphuretted hydrogen Yellow precipitate =

FILTRATE FROM AMMONIUM SULPHIDE.

Add hydrochloric in excess, filter, wash precipitate, and dry it. Divide into two parts.

(1) Boil with strong hydrochloric acid, filter into platinum dish or on foil, and add zinc · A black stain = *antimony.* Remove zinc, boil precipitate with strong hydrochloric acid, filter, and to filtrate add mercuric chloride. Grey or white precipitate = *tin.*

(2) Fuse with potassium cyanide and sodium carbonate in an ignition tube. Mirrors = *arsenic.*

287. Group III.—Precipitate from Ammonium Hydrate.—Treat with dilute hydrochloric acid and filter :—

Residue.

Wash and test a part in borax bead—

Blue bead = *cobalt.*
Red bead = *nickel.*

If a blue bead, dry the filter, ignite, dissolve ash in hydrochloric and nitric acid, almost neutralize with caustic potash, add potassium nitrate and acetic acid till acid, allow to stand, filter off yellow precipitate. To filtrate add caustic potash, filter, and test in borax bead for *nickel* as above.

Filtrate.

Add strong nitric acid, boil, almost neutralize with caustic potash; when cold add excess of barium carbonate and filter :—

Precipitate.

Wash and divide into 3 parts:—

(1) Dissolve hydrochloric acid, and add potassium sulphocyanate. A red colour = *iron.*
(2) Boil with solution of bleaching powder, and filter. A yellow filtrate = *chromium.*
(3) Boil with caustic potash, filter, to filtrate add ammonium chloride, and warm. White precipitate = *aluminium.*

Filtrate.

Add sulphuric acid, filter off barium sulphate, evaporate to small bulk, add excess caustic potash, and filter :—

Precipitate

Test in sodium carbonate bead for *manganese.*

Filtrate.

Pass sulphuretted hydrogen. White precipitate = *zinc.*

288. Group IV.—Precipitate from Ammonium Carbonate.—Dissolve precipitate in hydrochloric acid evaporate to dryness on water bath. Dissolve part of the residue in a little water, add calcium sulphate to the solution, and allow to stand.

If no precipitate is formed.—Dissolve rest of residue in water, add ammonium oxalate solution White precipitate = *calcium.*

If a precipitate is formed after some time = strontium Dissolve rest of residue in water, boil with ammonium sulphate and ammonium hydrate for some time, filter off precipitate, and to filtrate add ammonium oxalate. White precipitate = *calcium.*

If a precipitate is formed immediately = barium Digest rest of residue in alcohol, powdering it in a dish, filter, add sulphuric acid to filtrate, and filter. Boil precipitate with ammonium sulphate and ammonium hydrate for some time, and filter. —

Residue.	*Filtrate.*
Test for strontium on platinum wire in Bunsen flame · Brick-red flame = *strontium*	Dilute and add ammonium oxalate. White precipitate = *calcium.*

289. Group V.—Filtrate from Ammonium Carbonate.—To one part of filtrate add sodium phosphate solution, shake well, and allow to stand Crystalline precipitate = *magnesium*

If magnesium is absent—Evaporate rest of filtrate to dryness, ignite the residue on porcelain crucible lid till white fumes cease, dissolve residue in a *little* water, filter if necessary, into a watch glass, and test as below for potassium and sodium.

If magnesium is present—Evaporate rest of solution to dryness, ignite as above, warm residue in a little water, add milk of lime until alkaline, boil, filter, to filtrate add ammonium carbonate, warm gently, filter, evaporate the filtrate, dissolve residue in a *little* water, add a drop of hydrochloric acid, pour into a watch glass, and test as below for potassium and sodium

POTASSIUM AND SODIUM.

Dip a platinum wire into the solution in watch glass, and hold wire in a Bunsen flame A yellow colouration = *sodium.* Then add platinic chloride to solution, and stir Yellow precipitate = *potassium.* If no precipitate, evaporate to dryness on water bath, add a little water, and observe whether yellow powder remains undissolved. Warm original substance, with caustic soda or potash in a test tube, if *ammonia* is present it will be detected by smell.

Dry Reactions for the Metals.

290. Flame Test.—Dip a piece of platinum wire in strong pure hydrochloric acid, and heat the wire in the non-luminous flame of a Bunsen burner, until the wire glows, but imparts no colour to the flame. Now pick up a little of the substance to be tested on the heated wire, hold in the flame, and note the colour The colour reactions peculiar to the various metals will be found under the different headings

291. Borax Bead Tests.—Make a loop on the end of a piece of platinum wire, heat the wire to redness, dip the loop into a little powdered borax, and heat until a colourless bead is formed in the loop. To make the test, take up a little of the substance on the bead, and hold in the flame of the Bunsen burner. Notice the colour imparted to the bead. The characteristic colours distinguishing the metals will be found under the different headings

292. Charcoal Test.—Scoop out a hole in a block of charcoal In this place a little of the substance, and heat strongly in the blow-pipe flame. The behaviour of different salts will be found indicated in the tables included in this chapter.

293. TABLE OF ATOMIC WEIGHTS.

Element	Symbol.	Approximate Atomic Weight.	Exact Value.
Aluminium	Al	27·	27·04
Antimony	Sb.	120·	119 6
Arsenic	As.	75·	74·9
Barium	Ba.	137·	136·86
Beryllium	Be.	9·	9 08
Bismuth	Bi.	207·5	—
Boron	B.	11·	10·9
Bromine	Br.	80·	79·76
Cadmium	Cd.	112·	111·7
Calcium	Ca.	40·	39·91
Cœsium	Cs.	133·	132·7
Carbon	C.	12·	11·97
Cerium	Ce.	141·2	—
Chlorine	Cl.	35·5	35·37
Chromium	Cr.	52·	52 45
Cobalt	Co.	59·	58·6
Copper (Cuprum)	Cu.	63·	63·18
Didymium	Di.	145·	—
Erbium	Er.	166·	—
Fluorine	F.	19·	19·06
Gallium	Ga.	70·	69 86
Germanium	Ge.	72·	—
Gold (Aurum)	Au.	197·	196 8
Hydrogen	H.	1·	—

Element.	Symbol.	Approximate Atomic Weight.	Exact Value.
Indium	In.	113·	113·4
Iodine	I.	127·	126·54
Iridium	Ir	192·5	—
Iron (Ferrum)	Fe.	56·	55·88
Lanthanum	La.	138·5	—
Lead	Pb	207·	206 39
Lithium	Li.	7·	7·01
Magnesium	Mg.	24·	23·94
Manganese	Mn.	55·	54·8
Mercury (Hydrargyrum).	Hg.	200·	199·8
Molybdenum	Mo.	96·	95·8
Nickel	Ni.	59·	58·6
Niobium	Nb.	93 7	—
Nitrogen	N.	14·	14·01
Osmium	Os.	191·	—
Oxygen	O.	16·	15·96
Palladium	Pd.	106·	106·2
Phosphorus	P.	31·	30·96
Platinum	Pt	195·	194·3
Potassium (Kalium)	K.	39·	39·03
Rhodium	Rh.	104·	104·1
Rubidium	Rb	85·	85 2
Ruthenium	Ru.	103 5	—
Samarium	Sm.	150·	—
Scandium	Sc	44·	43·97
Selenium	Se.	79·	78·87
Silicon	Si	28·	28·3
Silver (Argentum)	Ag.	108·	107.66
Sodium (Natrium)	Na.	23·	22·995
Strontium	Sr.	87·3	—
Sulphur	S.	32·	31·98
Tantalum	Ta.	182·	—
Tellurium	Te	125·	—
Thallium	Tl.	203 7	—
Thorium	Th.	232·	—
Tin	Sn.	118·	117·35
Titanium	Ti.	48·	—
Tungsten	W.	184·	—
Uranium	U.	239·8	—
Vanadium	V.	239·8	—
Ytterbium	Yb.	173·	—
Yttrium	Y.	89·6	—
Zinc	Zn.	65	64·88
Zirconium	Zr.	90·4	—

NOTE.—The figures given in the third column are the values commonly used; the more accurate figures in the fourth column are employed in exact work.

294. CONVERSION OF TWADDELL HYDROMETER DEGREES.

Degrees Twaddell to Specific Gravity.—Multiply number of degrees × ·005, and add 1,000.

Specific Gravity to Degrees Twaddell.—Deduct 1,000, and divide the remainder by ·005.

295. CONVERSION OF TEMPERATURES.

Centigrade to Fahrenheit.—Multiply by 9, divide by 5, and add 32.

Fahrenheit to Centigrade.—Deduct 32, divide by 9, and multiply by 5.

296. COMPARISON OF WEIGHTS AND MEASURES.—

1 Metre = 3 280890 English feet.
1 Litre = 0 2201 gallons.
1 Hectolitre = 22·01 gallons
1 Kilogramme = 1,000 gm. = weight of 1 litre of water at + 4 C
= 2 2046 pound avoirdupois,
1 Gramme = 15·432 grains (English)
1 Quintal = 100 Kg = 196·84 lb. avoirdupois = 1 cwt. 3 qrs. 0·84 lb.
1 Metrical ton = 1,000 Kg. = 0·9842 English ton = 1 1023 American short ton (at 2,000 lb.)

1 Foot = 0 3047943 metre.
1 Inch = 25·3995 millimetres.
1 Yard = 0·9143835 metre.

1 Gallon = 4 quarts = 8 pints = 277·274 cub. inches = 4·536 litres
1 Cubic foot = 28·3153 litres.
1 Cubic inch = 16·3862 cubic centimetres.

1 Fluid ounce = $\frac{1}{20}$th pint = 28 35 cubic centimetres

1 Pound avoirdupois = 16 ounces = 7,000 grains = 0·4535926 kilogramme
1 Ounce avoirdupois = 437½ grains = 28·35 g.
1 Gallon = 10 lb. of water = 70,000 grains
1 Hundredweight = 112 lb. = 50·8024 kg.
1 Ton = 20 cwt. = 2,240 lb = 1016·648 kg.
Pound troy = 12 ounces troy = 96 drams = 288 scruples = 5,760 grains,
= 373·24195 gm.
1 Ounce troy = 8 drams = 24 scruples = 480 grains = 31·1035 gm.
1 Grain (common to avoirdupois and troy weight) = 0·06479835 gm.

WORKS OF REFERENCE

297. The following volumes are quoted as being the most useful where more exhaustive and complete methods of analysis are required than are included in the preceding chapters These works have been freely consulted in the compilation of this volume, and the author takes the opportunity of acknowledging his indebtedness The reference volumes may be regarded as standard works, and the reader cannot do better than acquire them, if he is desirous of forming a useful library of analytical literature:—

"Qualitative Analysis," Fresenius.

"Quantitative Analysis," Fresenius.

"Qualitative and Quantitative Analysis," Clowes and Coleman (J & A. Churchill).

"A Manual of Chemical Analysis, Qualitative and Quantitative," Newth (Longmans & Co)

"Quantitative Chemical Analysis," Thorpe (Longmans & Co).

"Volumetric Analysis," Sutton (J & A Churchill)

"The Alkali Maker's Pocket Book," Lunge & Hurter.

"Commercial Organic Analysis," Allen (J. & A Churchill).

"Gas Manufacture and the Assay of Bye-Products," Butterfield (Griffin & Co)

"Water Analysis," Wanklyn (Kegan, Paul, & Co.).

"The River Irwell and its Tributaries," Davis Bros., Manchester

"Painters' Oils, Colours, and Varnishes," Hurst (Griffin & Co).

"Lubricating Oils, Fats, and Greases," Hurst (Scott, Greenwood, & Co)

"Lubricants and Lubrication," Archbutt & Coleman

"Fuels. Their Analysis and Valuation," Phillips (Crosby, Lockwood, & Son).

1283

INDEX.

A

	Page No	Paragraph No
Abel-Pensky flash-point apparatus	70	129
Abel's flash-point apparatus	67	128
Absorbed oxygen in water, estimation of	138	235
Acid, free, in alum	97	184
Acid, hydrochloric	144	241a
Acid, nitric	145	241b
Acid, sulphuric	51	77—83
Acids, reactions of:—		
Borate	156	281
Chloride	156	277
Cyanide	156	279
Fluoride	156	282
Nitrate	156	278
Phosphate	156	280
Sulphate	156	276
Sulphide	157	283
Alkalinity in water, estimation of	138	234
Alum cake	97	182—184
Alum, estimation of alumina	97	182
Alum, estimation of free acid	97	184
Alum, estimation of insoluble matter	97	183
Alumina, sulphate of	97	182—184
Aluminium, reactions of	155	268
Ammonia, albumenoid, in water, estimation of	125	216
Ammoniacal liquor in coal tar, estimation of…	92	165
Ammonia concentrate, estimation of ammonia	89	155
Ammonia, free, in water, estimation of	125	215
Ammonia in gas liquor	88	153—158
Ammonia, sulphate of	87	151—152

	Page No	Paragraph No.
Ammonia, tests for	156	275
Analysis, qualitative	152	251—292
Analytical balance .	13	12
Anthracene in coal tar, estimation of	92	167
Antimony, reactions of	153	259
Apparatus for generation of sulphuretted hydrogen	53	82
Apparatus for volumetric analysis	30	27
Apparatus, selection of .	10	10—42
Apparatus, specific gravity	19	17
Apparatus, sundry	35	40
Apparatus, weighing . ..	10	10
Archbutt-Deeley water-softening plant	134	. 225
Arsenic, British Pharmacopœia tests for	151	249
Arsenic, detection of	147	246—250
Arsenic in coal and coke, estimation of	149	248
Arsenic in glycerine, British Pharmacopœia test for	151	250
Arsenic in sulphuric acid, detection of	52	81
Arsenic, Marsh's test for	148	246
Arsenic, reactions of	154	. 260
Arsenic, Reinsch's test for	148	. 247
Arsenic, Reinsch's test, modification of	148	247a
Atomic weights, table of	161	293
Available chlorine, estimation of	56	86—87

B

Balance, analytical . .	13	. 12
Balance, rough . .	12	. 11
Balance, use of . . .	15	. 14
Bale's apparatus for sulphur extraction	83	150
Barium, reactions of . ..	155	. 269
Basins, evaporating	35	38
Bath, sand . .	28	25
Bath, water . .	26	22
Beakers . ..	34	35
Beeswax, detection of Japan wax, tallow, etc	145	241c
Beeswax, detection of sulphur. . .	145	241c
Beeswax, estimation of mineral matter	145	241c
Beeswax, estimation of starch and flour .	145	241c

	Page No.	Paragraph No.
Beeswax, estimation of water	145	241c
Bink's burette	33	33
Bismuth, reactions of	153	255
Bleaching liquor, estimation of available chlorine	57	87
Bleaching powder, estimation of available chlorine	56	86
Bleaching powder, sampling	54	85
Bleaching powder, testing	54	84
Bog ore, estimation of hydrated oxide of iron	144	241
Bog ore, moisture in, estimation of.	144	240
Boiler fluids, estimation of alkalinity.	146	242
Brimstone, estimation of ash	63	108
Brimstone, estimation of moisture	63	107
Brimstone, estimation of sulphur.	63	109
Bunsen burners	27	24
Burette, Bink's	32	33
Burette, Mohr's	32	32
Burette, the	32	32
Burners, Bunsen	27	24
Burners, gas	27	23

C

Cadmium, reactions of	153	257
Calcium, reactions of	155	271
Calorific value of fuels	48	74
Calorimeter, Dr. Lewis Thompson's.	48	74
"Carrod" water-softening plant	134	225
Caustic liquor, estimation of available alkali	60	96
Caustic soda, estimation of available alkali	60	94
Caustic soda, estimation of insoluble	60	95
Caustic soda, sampling	60	93
Chemicals and raw materials used in paper making	93	169—205
Chemicals, miscellaneous	51	77—127
Chimney gases, estimation of hydrochloric acid	146	241d
Chimney gases, estimation of total acidity	146	241e
China clay, colour, etc.	95	171

	Page No	Paragraph No.
China clay, estimation of moisture	95	170
China clay, sampling	95	169
Chlorine in water, estimation of	124	214
Chromium, reactions of	155	267
Coal, calorific value of	48	74
Coal, estimation of ash	44	71
Coal, estimation of coke	44	70
Coal, estimation of moisture	43	69
Coal, estimation of sulphur	46	92
Coal, gasification value of	46	73
Coal, sampling	43	68
Cobalt, reactions of	154	263
Coke, calorific value of	48	74
Coke, estimation of ash	44	70
Coke, estimation of moisture	43	69
Coke, estimation of sulphur	46	72
Coke, sampling	43	68
Coleman-Archbutt viscometer, the	77	137
Copper, in water, detection of	128	222
Copper, reactions of	153	256
Crucible, Rose's	34	34
D		
Desiccator, the	17	16
E		
Effluents, trade	135	226—237a
Evaporating basins	35	38
F		
Fat, oil, or grease, in textile substances, estimation of	143	239
Filtering	36	43
Flasks	34	36
Flasks, measuring	30	28
Free acid in alum	97	184
Free acid in salt cake, estimation of	61	98
Fuel, liquid, calorific value of	49	76
Fuel, liquid, flash-point	49	75
Fuel, liquid, specific gravity of	50	76a

	Page No	Paragraph No.
Fuel, liquid, testing	49	75—76a
Fuels, calorific value of	48	74
Fuels, estimation of ash	44	70
Fuels, estimation of moisture	43	69
Fuels, sampling	43	68
Fuels, the testing of	42	67—76a
Funnels	35	37
Furnace, muffle	28	26

G

Gas burners	27	23
Gasification value of coal	46	73
Gas liquor, estimation of ammonia	88	154
Gas liquor, estimation of ammonia by direct titration	90	158
Gas liquor, estimation of specific gravity	88	153
Gas liquor, free and fixed ammonia in	90	157
Gas liquor, ounce strength	89	156
Gas residuals and bye-products	82	145—168
Gelatine, estimation of ash	96	176
Gelatine, estimation of moisture	96	175
Gelatine, relative strength	96	177
General reagents	41	66
Gray's flash-point apparatus	70	130

H

Hardness in trade effluents, estimation of	138	231—233
Hardness in water, estimation of	122	211—213
Hot-air ovens	25	21
Hydrochloric acid, detection of iron	145	241a
Hydrochloric acid, estimation of sulphuric acid	144	241a
Hydrochloric acid, percentage of HCl.	144	241a
Hydrochloric acid, specific gravity, estimation of	144	241a
Hydrometer degrees, Twaddell's, conversion of	163	294
Hydrometers, Twaddell's	19	17

I

Ignition of precipitates	36	45
Indicators	40	62—66
Indicators, indigo	41	65

	Page No	Paragraph No.
Indicators, litmus solution	40	62
Indicators, methyl-orange	40	63
Indicators, phenolphthalein	41	64
Iron in water, estimation of	126	217
Iron, reactions of	154	264

L

Laboratory, balance table	9	8
Laboratory, benches	8	4
Laboratory, burette table	9	7
Laboratory, fitting up	7	1
Laboratory, gas and water in	8	2
Laboratory, gas bench	9	6
Laboratory, shelves	8	3
Laboratory, sinks	10	9
Laboratory, water bench	9	5
Lead, in water, detection of	128	221
Lead, reactions of	152	253
Light oils in coal tar, estimation of	92	165
Lime, soluble, in water, estimation of	127	219
Limestone, detection of iron	65	120
Limestone, estimation of calcium carbonate	65	119
Limestone, estimation of insoluble matter	65	118
Linseed oil, estimation of specific gravity	80	140
Liquid fuels	49	75—76a

M

Magnesia, soluble, in water, estimation of	127	220
Magnesium, reactions of	155	272
Manganese, reactions of	154	265
Measuring flasks	30	28
Mercury, dyad, reactions of	153	254
Mercury, monad, reactions of	152	252
Metals, reactions of	152	251—274
Middle oil in coal tar, estimation of	92	166
Miscellaneous chemicals	51	77—127
Miscellaneous testing	143	238—292
Mohr's burette	32	32
Mortars	35	39
Muffle furnace	28	26

	Page No	Paragraph No
N		
Nesslerising	128	223
Nessler reagent for water analysis	40	60
Nickel, reactions of	154	262
Nitrate of soda, estimation of moisture	64	113
Nitrate of soda, estimation of insoluble matter	64	114
Nitrate of soda, estimation of nitrate	65	117
Nitrate of soda, estimation of sodium chloride	64	115
Nitrate of soda, estimation of sulphate of soda	65	116
Nitric acid, estimation of impurities	145	241b
Nitric acid, estimation of specific gravity	145	241b
O		
Oil, flash-point, apparatus for, Abel's	67	128
Oil, flash-point, apparatus for, Abel-Pensky	70	129
Oil, flash-point, apparatus for, Gray's	70	130
Oil, flash-point, a safe	73	132
Oil, flash-point of	67	128—132
Oil, flash-point test, simple	72	131
Oil, linseed, estimation of specific gravity	80	140
Oil, linseed, detection of adulteration with mineral oils	80	142
Oil, linseed, flash-point of	80	141
Oils, estimation of specific gravity of	79	137a
Oils for paint making	79	139
Oils, lubricating, comparison of samples	79	138
Oil testing	67	128—144
Oil, viscosity of	73	133—137
Oil, viscosity of, by pipette method	73	133
Oil, viscosity of, method of expressing results	76	136
Ovens, hot-air	25	21
Ovens, water	23	20
P		
Paper, estimation of ash	98	187
Paper, estimation of moisture in	98	188
Paper making, chemicals and raw materials used in	93	169—205
Paper, mechanical wood pulp in, detection of	98	190

	Page No	Paragraph No.
Paper, physical testing of	98	189
Paper testing	98	187—190
Pearl hardening, estimation of total moisture	97	185
[Paragraph 185 should read, "*heat in the hot-air bath.*"]		
Pearl hardening, comparison of colour	97	186
Pipettes, bulb	30	29
Pipettes, scale	31	30
Pipette, the	30	29
Pipette, use of	32	31
Pitch in coal tar, estimation of	92	168
Platinum vessels, precautions to be observed in using	36	44
Potash, chlorate of, estimation of chloride	66	127
Potash, chloride of, estimation of moisture	65	121
Potash, chloride of, estimation of total chloride	65	122
Potash, sulphate of, estimation of free acid	66	123
Potash, sulphate of, detection of iron	66	126
Potash, sulphate of, estimation of insoluble matter	66	125
Potash, sulphate of, estimation of potassium chloride	66	124
Potassium, reactions of	155	273
Precipitates, ignition of	36	45
Prussian blue in spent oxide	90	159
Pulp, wood, sampling and testing. (See "Wood pulp.")	98	191—205
Pyrites, burnt, estimation of copper	60	91
Pyrites, burnt, estimation of moisture	60	92
Pyrites, burnt, estimation of sulphur	60	90
Pyrites, estimation of copper	59	89
Pyrites, estimation of moisture	59	88a
Pyrites, estimation of sulphur	58	88

Q

Qualitative analysis	151	251—292
Qualitative analysis, borax bead tests	161	291
Qualitative analysis, charcoal test	161	292
Qualitative analysis, flame test	161	290
Qualitative analysis, tables for	157	284—289

	Page No	Paragraph No
R		
Raw materials, necessity for testing	1	
Reactions of the acids . .	156	276—283
Reactions of the metals . . .	152	251—274
Reagents, general	41	66
Recovered soda, estimation of available alkali	96	174
Redwood's viscometer . ..	74	134—135
Riddell filter	130	224
Rose's crucible .	34	34
S		
Salt cake, estimation of free sulphuric acid	61	98
Salt cake, estimation of insoluble matter	61	99
Salt cake, detection of iron. .. .	61	100
Salt cake, estimation of sodium chloride	61	97
Salt cake, estimation of total sodium sulphate	61	101
Salt, estimation of insoluble matter . .	64	111
Salt, estimation of moisture. . . .	64	110
Salt, estimation of sodium chloride	64	112
Sand bath	28	25
Selenium, test for....	154	261
Silica in water, estimation of	127	218
Silver, reactions of	152	251
Soap, estimation of free alkali. . . .	147	245
Soap, estimation of unsaponified fat .	146	244
Soap, estimation of water	146	243
Soda ash, estimation of available alkali	63	106
Soda ash, estimation of insoluble matter	62	105
Soda ash, sampling	62	104
Soda, caustic	60	. 93—95
Soda crystals, estimation of available alkali	62	103
Soda crystals, estimation of insoluble matter	62	102
Soda, recovered, estimation of available alkali	96	174
Sodium nitrate. (See nitrate of soda) .	64	113—117
Sodium, test for	156	274
Solutions, standard.	37	46
Specific gravity apparatus.	19	. . 17
Specific gravity balance, Westphal	20	. 18
Specific gravity bottle	22	. 19
Spent oxide	82	145

	Page No.	Paragraph No.
Spent oxide, estimation of moisture	83	147
Spent oxide, estimation of Prussian blue	90	159
Spent oxide, estimation of sulphur	83	148—150
Spent oxide, sampling	82	146
Standard solutions .	37	46
Ammonium chloride solution, for Nesslerising . ,	39	56
Arsenious acid solution, for available chlorine .	39	53
Calcium chloride solutions, for hardness in waters	40	59
Caustic alkali, normal	39	51
Hydrochloric acid, normal	38	49
Iron solution	40	61
Nitric acid, normal	39	50
Oxalic acid, normal	39	54
Potassium permanganate, alkaline, for water analysis	39	57
Potassium permanganate, deci-normal	39	52
Soap solution ..	39	58
Silver nitrate, deci-normal	39	55
Sodium carbonate, normal	38	47
Sulphuric acid, normal	38	48
Starch, estimation of ash	96	179
Starch, estimation of insoluble matter	97	181
Starch, estimation of moisture	96	178
Starch, relative strength .	97	180
Strontium, reactions of	155	270
Sulphate of alumina	97	182—184
Sulphate of ammonia, estimation of ammonia	87	152
Sulphate of ammonia, estimation of moisture	87	151
Sulphuretted hydrogen apparatus	53	82
Sulphuric acid .	51	78
Sulphuric acid, detection of arsenic	52	81
Sulphuric acid, detection of iron	52	80
Sulphuric acid, detection of lead	52	79
Sulphuric acid, detection of nitrous acid	53	83
Sulphuric acid, real sulphuric acid in	51	78
Sulphuric acid, specific gravity of	51	77
Sulphur in coal, estimation of .	46	72

	Page No	Paragraph No
Sulphur in coke, estimation of	46	72
Sulphur in spent oxide	83	148—150
Sulphur (See brimstone)	63	107—109
Sundry apparatus.	35	40
T		
Tar, estimation of ammoniacal liquor	92	165
Tar, estimation of anthracene .	92	167
Tar, estimation of light oils	92	165
Tar, estimation of middle oils	92	166
Tar, estimation of pitch	92	168
Tar, examination of	91	164
Temperatures, conversion of	163	295
Testing of fuels.	42	67—76a
Textile substances, estimation of fat, oil, or grease	143	239
Textile substances, estimation of moisture	143	238
Thermometers .	35	41
Thompson's calorimeter	48	74
Tin, reactions of .	153	258
Trade effluents .. .	135	226—237a
Trade effluents, estimation of absorbed oxygen	138	235
Trade effluents, estimation of alkalinity	138	234
Trade effluents, estimation of organic matter	138	230
Trade effluents, estimation of permanent hardness	138	232
Trade effluents, estimation of solids in solution	138	229
Trade effluents, estimation of suspended matter .	137	227
Trade effluents, estimation of total hardness	137	231
Trade effluents, estimation of total solids	137	228
Trade effluents, official report on ..	141	237
Trade effluents, purification of.. .	142	237a
Trade effluents, sampling	137	226
Trade effluents, testing against river inspectors	139	236
Turpentine, flash-point of . .	81	144
Turpentine, specific gravity of	8	143
Twaddell's hydrometer degrees, conversion of	163	294
Twaddell's hydrometers . . .	19	17

	Page No	Paragraph No.
V		
Viscometer, Coleman-Archbutt, the	77	137
Viscometer, Redwood's	74	134—135
Viscometer, the	74	134—137
Volumetric analysis, apparatus for	30	27
W		
Wash bottle	35	42
Washing soda	62	102—103
Water analysis	117	206—223
Water analysis —		
Alkaline permanganate solution for	39	57
Alkalinity, estimation of	138	234
Ammonia-free water	118	206
Calcium chloride, solution for	40	59
Copper, detection of	128	222
Estimation of absorbed oxygen	138	235
Estimation of albumenoid ammonia	125	216
Estimation of chlorine	124	214
Estimation of dissolved solids	122	209
Estimation of free ammonia	125	215
Estimation of permanent hardness	123	212
Estimation of suspended matter	121	208
Estimation of temporary hardness	124	213
Estimation of total hardness	122	211
Estimation of total solids	121	207
Iron in solution, estimation of	126	217
Lead, detection of	128	221
Loss on ignition, estimation of	122	210
Nesslerising	128	223
Nessler reagent for	40	60
Silica, estimation of	127	218
Soap solution for	39	58
Soluble lime, estimation of	127	219
Soluble magnesia, estimation of	128	220
Standard ammonium chloride solution for Nesslerising	39	56
Standard iron solution for	40	61
Water bath	26	22
Water ovens	23	20

	Page No	Paragraph No.
Water purification and filtration	130	224
Water softening	133	225
Water-softening plant, Archbutt-Deeley system	133	225
Water-softening plant, "Carrod" system	133	225
Weighing apparatus	10	10
Weighing, directions for	17	15
Weights	14	13
Weights and measures, comparison of	163	296
Westphal specific gravity balance	20	18
Wood pulp —		
Drying ovens	109	197—200
Mechanical, in paper, detection of	98	190
Sampling and testing of	98	191—205
Testing, air-dry pulp in parcel	114	203
Calculation of results	114	202
Damaged bales	100	193
General rules for sampling and testing	115	205
Method of sampling	106	196
Oven, air	110	199
Oven in which sample is weighed in drying chamber	111	200
Oven, water	109	198
Percentage of bales to be sampled	99	192
Selection of bales	105	194
Uniformity in sampling and testing	114	204
Use of the dessicator	113	201
Weighing bales	105	195
Works of reference	164	297

Z

	Page No	Paragraph No.
Zinc, reactions of	154	266

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Speciality Boiler Fluid, for all Household Boilers where a circulation of hot water is set up, also where water is used for hot baths, washing, etc New Boiler Scale Vegetable Fluids

New Speciality Boiler Composition, Extraordinary Strength. Has stood the most severe tests for 25 years, superior to the largely-advertised brands, free from all injurious ingredients. Will soften and remove scale (however thick), thus preventing necessity of chipping, so often injurious to Boilers. Numerous Testimonials

American Boiler Fluids and Compounds, preventing Incrustation, etc.

"Cambrian" Boiler Compound.

Dilnutt's Anti-Corrosive Boiler Preparation, free from all acids. Does not affect the water or steam in any way, and can be used for all purposes. It has been vigorously tested and used by the leading steam-power users throughout the country, and universally admitted to be the Most Effectual Preparation for its purpose; also adapted for either Stationary, Locomotive, or Marine Boilers.

Directions for Use—¼ pint per horse power once a week. Made in three strengths and qualities, Nos. 1, 2, and 3. Costs few pence per gallon, and sells at 4s. per gallon.

Fossil Meal Compo Covering. Costs £5 per ton.

Asbestos Covering. Costs £4 per ton.

Speciality "Crown" Covering. Costs £1 per ton.

Special Tannic Compos., Powders, or Liquids. Preserves plates and prevents incrustation, causes precipitation of the salts in the water in form of soft mud, which can be easily washed out, and prevents hard scale in boilers.

New Continental and American Boiler Purges and Anti-Crustators, for all kind of boiler or waters.

Latest Improved Processes for Tannate of Soda, for preventing and removing incrustation and corrosion of Portable, Locomotive, Marine, or any kind of Steam Boilers.

Anti-Petrifying Mixture, for Incrustation of Boilers, etc

Latest Improved English and Foreign Engine Packings

Special Black Insulating Mixture for outside of Electric Cables.

Insulating Mixture for Covering Electric Tapes

Latest Inventions and Specialities in Belting Creams, Syrups, Dressings, etc, New Soluble Disinfectant Belting Syrup.

Preservatives for all kinds of Beltings—Hair, Rubber, Link, Cotton, or any kind—for working in or out of doors, or in water Will stand any climate, preventing slipping, giving more speed and splendid gripping power It penetrates the leather, tightens slack belts, waterproofs the leather, and will not crack.

New "Sensitive" Paint, changes from yellow to dark red at 120° Fahr, for painting Bearings, Boilers, Machinery, Hot-water Pipes, Electric Installations, etc ; returns to yellow colour, on cooling.

HOUSEHOLD SOAPS.

Latest Improved Recipes, New Processes, and Specialities.

Household, Mottled, and Grained Soaps in all kinds and qualities

Special Pale Yellow Bar Soaps Fine quality, double extra, etc Imperial Gold Pale, Best Pale, First Extra Pale, Best Wax Pale, Prime Extra Pale, 2nd Pale, Genuine Tallow Double Extra, Primrose, Pale 2nd and 3rd Qualities, etc. Special X, XX, and XXX Yellow Bar, etc, etc. New Household Soaps by latest English and Foreign Methods Speciality Soaps for Export to all Climates, etc.

Fireproof Household Soaps, rendering all fabrics washed with same non-inflammable and absolutely fireproof, and at same time cheapening the Soap.

Fine Pale and Primrose Soaps, Extra Pale Crown Soaps.

Special Curd and Mottled Soaps, Imperial, Extra, etc., etc.

New Pale Household Soaps, etc., etc.

Speciality Soaps like "Sunlight," "Titan," "Ivy," "Sun," "Aurora," "Lightning Cleanser," "Sunbeam," "Watson's," "Lawson's," "Matchless Cleanser," etc., etc.

Special Cotton Oil Soaps.

Cheap Bar Soaps, costing from about £5 per ton

New Cleaners and Speciality Soaps, as largely advertised

Special White Soaps for Laundries

Superior Washing and Cleansing Fluids, Compounds, etc.

New Quick Process for Soap Making, no boiling

Latest Methods for making Soaps by Cold Process.

Latest Methods for Lye, to give fine gloss to Surface of Soaps.

New Fillings and Cheapeners for large yields of Soaps.

Carpet Soaps

Carriage, Saddle, and Leather Soaps.

American Harness Grease, Oil Soaps, softens and preserves. Best Compo for Harness, giving Jet Black Lustre; also for Brown Leathers, etc. (Waterproof).

Sanitary Soaps (Carbolic, Creosote, Ammonia, etc)

Special Liquid Sanitary Soap.

Fine Disinfectant and Antiseptic Soaps (Special Cheap Qualities).

New Marine Soaps, special cheap qualities. White, etc.

Dye Soaps, like "Maypole," "Rainbow," etc, all Shades and Colours.

New Dye Soap (all Colours), Simple process, no Anilines.

Bone and Wax Soaps.

Resin Soaps. Special Qualities up to 50 per cent. of Resin

Castile and Marseilles Soaps. Frictional and Hydrated Soaps.

Cheap Soaps from Wastes and Refuse.

Cheapened Oils and Fats for Soap Makers.

Speciality new simple process for recovering Glycerine from Spent Soap Lye. No extra plant required.

Pure Glycerine and Recovered Glycerines by latest Processes.

New Special Method for Treating all kinds of Wastes and Refuse Greases for Soap Makers, enabling good quality Soaps to be made at about half the usual cost.

New Cheap Material for Soap Makers, equal to Best Pure White Tallow.

New Process for Bleaching Dark "Nigre."

New Process for Bleaching Cotton Seed Mucilage for making cheap Soaps, equal to Best in the Market.

POLISHING SOAPS IN ALL KINDS AND QUALITIES.

Sand Soaps—Silver Soaps.
Special Soaps like "Sapolio," "Monkey Brand," etc.
New Cheap Polishing Tablets for Jewellery, Metals, etc.
Special Silver Soap

MANUFACTURERS' SOAPS.

In all kinds and qualities and for all purposes.
Latest Recipes, Improved Methods, and Specialities.

Milling and Fulling Soaps.

Fireproof Manufacturers' Soaps, rendering all fabrics non-inflammable and absolutely Fireproof, and at same time cheapening the Soap.

Speciality Soaps for Silks and printed Goods

Speciality Soaps and Detergents for Calico Printers.

Sizing Soaps (Hard and Soft) for Cotton Manufacturers.

Softening Soaps for Cotton Manufacturers.

Cop Bottom Soap for Cotton Manufacturers and Spinners.

Bleachers' Scouring Soaps and Bleaching Soaps for all purposes

Warp Finishing Soap

Pure White Oil Soaps for Silks and Woollens

New Wool Washing Soaps and Liquids

Pure Potash Wool Scouring Soaps

New Washing and Scouring Liquids for Woollens, Silks, Linens, etc. No rubbing

Speciality Dry Soap Powder for Wool Scourers, etc.

Speciality Soaps for Dyers, Tanners, and Paper Makers

Manufacturers' Hard Scouring Soaps from Oil Foots, Yorkshire Grease, etc Sixty to 65 per cent. Fatty Acid.

Manufacturers' Pure Oil Soaps and Neutral Hard Soaps.

Special Manufacturers' Scouring Soaps.

Cheap Washing and Scouring Fluids for all Manufacturing Purposes.

Latest Cold Process Manufacturers' Soaps.

Special Moist Curd Soap for Silk Dyers, etc.

Benzine Soaps for Dyers and Cleaners

Special Naphtha Soft Soaps for Cleaning Delicate Fabrics, Silks, Colours, etc.

Special qualities suitable for any market & for all Trade purposes.

Speciality White Soft Soap for Dyers.

Bleaching Liquids for all purposes.

Speciality Condenser Detergents for Cleaning Marine Condenser Tubes, etc

DRY SOAPS, EXTRACTS, AND POWDERS.

New Recipes, Improved Processes, and Specialities.

Dry Soaps and Extracts in all kinds and qualities. equal to the largely-advertised Brands costing from 3s. 6d per cwt.

Fireproof Dry Soaps and Extracts, rendering all fabrics non-inflammable and absolutely fireproof.

Special Dry Soap, 25 per cent superior to any in the Market.

New Dry Soaps, Musk, Paraffin, Carbolic, Ammonia, etc., etc.

Speciality Musk Paraffin Dry Soaps, like "Moulton's"

New Cheap Artificial Musk (perfection); also Artificial Otto of Roses

Speciality Perfumes for Dry Soaps and Extracts

New Improved Dry Soaps and Extracts for Laundries.

American Concentrated Soap Extract for Laundries

New American Dry Cleaning Preparation No water used, like "Marvello" Compound.

Speciality American Dry Soaps, like "Babbitt's"

Special Self-cleansing Dry Soap

New Improved Oleine Dry Soap.

Hygienic Dry Soap. High Glaze for Calico Printers and Finishers. Speciality Dry Soaps, like "Hydroleine," for Laundries, Wool, Hosiery, and Leather Trades; also for Dyers, Furriers, Carpet Cleaners, Silk and Lace Washers, etc. Softens the hardest water, and can be used with sea water. Will not injure the finest fabrics and most delicate colours. Splendid disinfectant, and will wash clothes without rubbing; no injurious chemicals, only absolutely harmless ingredients. No Common Soda Base. Washes linen with snowy whiteness. A universal cleansing preparation.

SOFT SOAPS.

Latest Improved Recipes, New Processes, and Specialities.

Latest English, Scotch, Continental, and American methods for making all kinds and qualities of Soft Soaps for all purposes.

Manufacturing can be commenced at once from our Recipes.

Figged Soaps, Sanitary Soft Soaps, Carbolic Ammonia, New Household and Manufacturers' Soft Soaps, all kinds and qualities, etc., etc.

Naphtha Soft Soap.

Soft Soaps, no boiling (Speciality).

New White Semi-soft Soap for Laundries and Manufacturers. Superior to any Dry Soap or Extract, and perfect disinfectant

Medicinal and Borax Soft Soaps

Cheap Common Soft Soaps for all purposes.

New Improved Heavy Fillings for Soft Soaps

Genuine Transparent Soft Soap 134, 136, 142 firkins from one ton oil. Warranted to keep

New Linseed Oil Soft Soap, cost £8 to £8 10s. per ton.

Speciality Soft Scouring Soaps for Manufacturers.

Special Odourless Soft Soaps, for manufacturers' and scouring purposes, wool combers, silk and lace washers, etc Pure vegetable oils and potash Costing from £7 per ton, like "Savonal," etc.

Benzine Soft Soaps, and Liquid Soft Soaps, for dyers, cleaners, etc.

Speciality Soft Soaps for Toilet and Domestic Purposes

New quick process for making Transparent Odourless Soft Soaps.

High-class Soft Soaps for all purposes.

New Green Odourless Soft Soap, no colouring matter

New Resin Soft Soaps.

New Soft Soap Fillings, 20 to 35 per cent can be added, greatly improving the transparency of the soap

Cheapened Oils, etc, for Soft Soap Makers

Fireproof Soft Soaps, rendering all fabrics non-inflammable and absolutely fireproof, and at the same time cheapening the soap.

New Cheap, Quick Methods for making Improved Soft Soaps in 1½ to 2 hours

New Speciality Soft Soaps 3½ tons of first-class soap from one ton of oil

New Soft Soaps, made from Speciality Dry Soap

Pure Linseed Oil Soft Soaps 130 firkins from one ton of oil.

Cheap Pure Linseed Oil Soft Soaps, warranted firm and clear. Cost £6 10s per ton

Pure White Speciality Soft Soaps.

New Cheap Soft Soaps, made from Wastes, Refuse, etc

Speciality Soft Soaps for Temperate, Hot, and Cold Climates.

New Transparent Soft Soaps by improved processes.

Speciality Ammonia Semi-soft Soap.

Pure White Soft Soap for Dyers.

Special Naphtha Soft Soap for Cleaning delicate Fabrics, Silks, Colours, etc

New Soft Soaps by Cold Process.

American White Figged Soft Soap.

Special Green Soft Soaps, Nos. 1 and 2

Hempseed or Imitation Olive Oil Soft Soap

Speciality Odourless Soft Soaps (large yields)

Sanitary Soft Soaps in all kinds and qualities

Latest Improved Recipes for making all kinds of English and Foreign Household and Manufacturers' Soaps, New Soap Powders and Extracts, Washing Liquids, Laundry Specialities, New Soft Soaps (large yields). Latest Toilet Soaps and High-class Specialities Guaranteed same as worked from by the leading firms, and supplied to the various Governments and largest Contractors, including Prize and Gold Medal Brands, etc, and equal and superior to the largely-advertised specialities Any Samples matched and Working Recipes supplied.

DISINFECTANTS, SHEEP DIPS, WEED KILLERS, &C.

New Recipes, Inventions, and Latest Specialities.

Latest Improved Disinfectants, Powders, Fluids, Pastes, Sizes, Creams, Paints, Soaps, Compositions, etc, in all kinds and qualities

Pink Naphthaline Disinfectant Powder No Lime Base and no Colouring. Cost 1s. 6d per cwt

New Soluble Disinfectant Powder.

Extra Powerful Disinfectants and Deodorizers

New Disinfectant Sawdust

Special Improved Disinfectant Powder. Cheaper than Carbolic. No Lime Base, etc. Non-poisonous and non-corrosive.

Special Disinfectant Fluids and Oils, like "Jeyes," "Sanitas," "Pinol," "Kreasole," "Microzol," "Carbolezene," "Condy's," "Puritas," "Healthitas," etc., costing from 3d. per gallon. Latest improvements.

Improved Soluble Creosote

High-class Disinfectant Fluid for Medicinal Purposes, like "Creolin."

Colourless Disinfectant Fluid. Cost 1½d. per gallon.

New Antiseptic Fluids.

Eucalyptus Fluid. Costs 2½d. per gallon; sells at 6s. per gallon.

Special Eucalyptus Disinfectant Powder Costs 1s 6d. per cwt

White Carbolic Acid.

Special Disinfectant Fluid, like "Cresyphenol"

New Disinfectant Fluids, Powders, and Sheep Dips, like "Carboline."

New Improved Cold Process for Concentrated Creosate Disinfectant Fluids and Sheep Dips Non-poisonous, no Plant or Machinery required, materials simply mixed together in any Tub, Vat, or Tank In all qualities costing from 4d per gallon (no Resin), and equal to most of the leading Brands in the Market.

Artificial Carbolic Disinfectant Fluid. Quite as powerful as the genuine.

New Artificial Carbolic Acid (undetectable)

Special Carbolic Pine Odour Disinfectant (non-poisonous).

Sanitary Crystals, Shields, and Compounds (all Colours)

New American Disinfectants, like "Chloro-Naphtholium"

Cloudy Domestic Ammonia, like "Scrubbs." Cost 4d per gallon.

American Antiseptic and Disinfectant, like "Listernia"

Disinfectant Tablets for Urinals

New Solidified Disinfectant, rapidly miscible in water Splendid Deodorant for placing in Cisterns of W C's, etc.

Soluble Disinfectant Beltings.

Special Poisonous and Non-poisonous Weed Killers.

SHEEP DIPS AND VETERINARY SPECIALITIES.

Poisonous and Non-poisonous Fluid Sheep Dips One gallon makes 100 gallons dip, equal to the largely-advertised Specialities.

Speciality Non-poisonous Sheep Dips for Export. Soluble in all Waters

New Solidified Paste Sheep Dips, like "McDougall's," "Quibell's," etc.

Glycerine Sheep Dips

New Sulphur-Arsenical Paste Dips Perfectly Soluble

Speciality Sodio-Sulphide of Arsenical Powder Sheep Dips.

Improved Genuine Powder Dip, like "Cooper's"

Sheep Dipping Compounds, like "Premier"

Special Carbolic Sheep Dips for Export.

New Improved Disinfectant Foot-Rot Pastes, Lotions, etc

Latest Fly Washes and Powders.

New Fly Oil, Instantaneous Maggot Killer and Wound Healer.

Disinfectant Veterinary Ointments.

Latest Insect Powders, for all purposes

Our Recipes are equal to the largely-advertised Specialities.

NEW UNPATENTED RECIPES, INVENTIONS, AND SPECIALITIES.

Laundry Blues, Squares, Liquids, etc.

Speciality Blue, like "Reckitt's."

High-class Starch, like "Reckitt's," by latest and most scientific methods of production, to arrive at which some hundreds of pounds have been spent; only in the hands of very few firms, Success guaranteed. Erection of plant and machinery to successfully produce from ½ ton to 60 tons per day. Also Rice, Maize, and Wheat Starch, etc

Fireproof Starch, rendering all fabrics non-inflammable and absolutely fireproof

Starch, Enamels, and Glazes, like porcelains

New Concentrated Starches and Double Starch.

Speciality Glossy Starches

New Liquid Starch Glazes.

Patent Flour and Crystal Starches, having fine gloss.

Starch Gloss, Lump, Powder, Blocks, like "Mack's," etc

New Soluble Starch Gloss (Powder), Perfection

Waterproofings and Preservative Solutions for Fabrics, and Canvas Bags for all purposes.

Tinting Blue for Bleachers.

Tinting Paste for Bleachers

Chinese Blue for Dyers, Calico Printers, etc

Special Prussian Blue for Paper Stainers and Calico Printers.

Cattle and Veterinary Specialities.

Latest English and Foreign Veterinary Oils, Embrocations, Liniments, and Preparations

New Specialities Superior to any in the Market.

Embrocation similar to Elliman's.

Artificial Camphor, cannot be detected from the genuine article for all general purposes.

"Metallizene," a Composition for Refining Steel, which by simple process of plunging the hot metal when in course of manufacture, and afterwards working up into articles required, will impart such properties as make it tougher and more durable than it was originally, transforming it into the finest silver steel. It will also restore the properties of steel when destroyed or burnt in process of manufacture, and will enable any practical smith to convert the commonest quality into the best for all useful purposes All tools can be treated and become much tougher and more durable.

Speciality Black Harness Oil or Reviver. Costs 8d. per gallon; sells at 4s. per gallon. A perfect substitute for all blackings, compos, and pastes, softens, preserves, and produces a jet black to all leathers. Will clean harness ready for use in ten minutes. In universal demand by all leading firms, large carriers, and contractors, railway, tram, and omnibus companies, and all extensive users and large horse owners. The best article ever used in the stables.

New Speciality Jet Black Harness Oil Costs 11d per gallon No mineral oil.

Bleacher, like "Parazone," for linen and cotton goods

Improved Bleaching Liquids for all purposes

Calico Printers' Preparations.

Albumen Substitute or Albumenoid for Calico Printers

American Blood Albumen Pastes.

Tartarline, perfect substitute for Cream of Tartar

Artificial Beeswax Costs 2d. and 2½d. per lb. Pale, Dark, or White.

New American Brilliant Whitewash

New Washable "Whitewash"

New Speciality Insect Powders.

Brewers' Finings, Headings, Yeasts, Food Colourings and other Chemicals for Brewers, Distillers, and Wine Merchants

New Perfect Paste, superior to "Stickphast." Warranted to keep in all climates.

New Preparation, like "Gloy"

New Cheap Baking Powders.

Blanc Mange Powders. All flavours, shades, and tints (harmless colourings).

Table Jellies.

Pudding Powders.

Gilding and Silvering Glass, etc., by latest methods

Incombustible and Waterproof Paper.

Iron Filling Composition.

Substitute Composition for Window Glass for Roofings, etc.

Copper Coating Solution for Wires, Metals, etc.

Printers' Roller Compositions and Utilising Old Composition.

Photographers' Materials

Dyes and Dyers' Materials

Speciality Window Cleaning Paste, produces fine lustre and prevents steaming

Fire Extinguishing Solutions and Preparations

Preservatives for Foods, Game, Fish, Milk, etc.

Grease Eradicator, odourless, equal to Benzine.

American Ball-room Powder.

New Ball-room Polish, will make the roughest flooring smooth like ice.

New Substitute for Emery.

Speciality Curtain Tints, like "Dolly," "Ripley's," etc.

Insulating Gum.

Waterproofing Solutions and Compounds for all purposes.

New Waterproofings for Silks, Canvas, and all Fabrics.

Speciality American Waterproofing, costs about 2s. for 60 gals.

New Process for Manufacturing Aniline Dyes (all colours) from Anthracene (cheaper than German methods).

New Substitutes for Turpentine No Still or Plant required—simply mixed cold. Cost from 6d. per gallon.

Deodorised Petroleum, Kerosene, Benzine, Naphtha, Turps, etc., by new cheap American process. Cost about 4d. per barrel Absolutely odourless

Fireproof (Colourless) Solution, rendering all fabrics, paper, etc, non-inflammable and absolutely fireproof.

Soda Manufacture by latest improved methods

Sand Paper by latest methods in all kinds and qualities.

Veleuring Compos for Hat Manufacturers.

Ammonia—Fort, Fortis, Anhydrous, pure ·880.

Sulphate, Nitrate, etc, etc.

Coloured Ammonia, Heliotrope, Pink, Green, Blue, etc. Permanent Colours.

Extract of Indigo Liquid Paste.

Refined Indigo.

Pure Indigotine.

Nitric Acid, with Efficient Method for Original Colourless Acid

Nitrate of Lead, for Practical Commercial use

Cudbear and Paste Archill; also Liquid Archill—both from Orchella Weed—noted brand.

Persulphate of Iron; sold as Nitrate of Iron.

Latest Improved Methods for Acetic Acids

Carbolic Acids in all qualities, Crystallised Carbolic Acids, etc.

New Improved Process for Concentrated Sulphuric Acid.

Muriatic Acid.

Arsenic Acid.

Method for safely emptying sulphuric or other acids, any quantity, no loss or damage.

Acid Solutions, and all kinds of Chemicals a Speciality

Dipping Acids, Soldering Spirits, etc.

Improved Process for Refining Saltpetre

Sulphate of Soda (Glauber Salts).

Hydrochloric Acid (Chemically Pure).

Picric Acid.

White Leads by latest English and Foreign Processes.

Acetate of Lead (White Sugar of Lead).

Cyanide of Potassium (commercial for electro plates, etc.).

Methylated Spirits. Heavy and Fine Chemicals for all purposes.

Any Samples Matched, and Working Recipes Supplied.

New Fly Papers—Sticky or Dry (Non-Arsenical, etc)

New Sticky Fly Compositions, like "Marshall's."

Celluloid, clear as glass. Perfectly resists all Acids, Salts, lkalies, etc

Crystallising on Tin Plates, all colours. New Glazes for Bricks.

Enamelling on Iron, latest improvements

Incombustible and Waterproof Papers, Cardboards, etc.

Greaseproof and Waterproof Compositions for Paper

Iridescent or Marbled Paper (all Colours)

Paper and Papier Maché Manufacture by latest English and 'oreign Processes

Paper Makers' Materials, Improved Pulps, Gelatines, Sizes, anitary and Distemper Colours, etc.

Photographers' Materials, Composition, Varnishes, Preparaons, etc, by latest Processes. Dyers' Materials, Sizes, New 'yes, etc.

Matches by latest English, Continental, and American Proesses; New Improved Compounds, Anti-phosphorous, Extra afety, Non-explosive, Inextinguishable, etc, etc

Tanners, Curriers, Fellmongers, and Leather Dressers [aterials, Compositions, Preparations, etc.

Latest English and Foreign Processes for Unhairing Hides, [ew Solvents (no Caustic), etc.

Utilisation of Wastes and Refuse, Improvements in Bi-products, c, by latest Processes.

NEW SPECIALITY RECIPES (UNCLASSIFIED).

New Process for making any Cheap Resins perfectly White ıd Hard for Varnishes, etc

New Compo for making Incandescent Mantles flexible.

Compositions and Paints, like "Ferrodor."

New Asbestos Compositions New Paints similar to "Bells" sbestos Aquol Paint, fire and water resisting, non-poisonous, etc.

New Enamels, like "Vitros" or Liquid Porcelain.

Improved Process for degumming Rhami Grass for Manufacıring

Government Waterproof Floor and Roofing Paints.

New Process for cheapening Tallows up to 50 per cent. of ıter.

New Process for making Cotton Oils equal to Olive Oils.

Improved Process (Hot or Cold) for treating all Linseed Oils solutely free from spawn and impurities for varnish making and ırranted not to break up to 700° Fahr, perfectly de-albumenised.

New Simple Process for Seed Crushing greatly improves lour of the Oils and largely increases profit for the Oil Cakes.

Our Recipes are worked from by the leading Firms.

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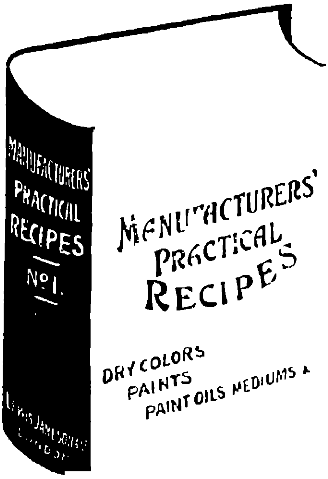
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MANUFACTURERS' PRACTICAL RECIPES
DRY COLORS
PAINTS
PAINT OILS MEDIUMS &

IN THE PRESS.

BOOK No. 1.

MANUFACTURERS' PRACTICAL RECIPES

A Treasury of Original Recipes and Processes.

INCLUDING

Latest Improved Up-to-Date English, Continental and American Recipes and Processes, with full Working Instructions for Manufacturing

Dry Colours,
Distemper Paints,
Stiff and Paste Colours,
Substitutes for Linseed Oils,
Paint Mediums,
Paints,
Silicate Paints,
Putty,
Turpentine Substitutes,
Chemical Colours,
Paint Oils,
&c., &c , &c.

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MANUFACTURERS' PRACTICAL RECIPES.

BOOK NO. 1.

DRY COLOURS, PAINTS, PAINT OILS, PAINT MEDIUMS, &c.

CONTENTS:

PART I.—Pigments and Dry Colours.

CHAP. I —Natural Colours and their Derivatives.—Red Oxide of Iron; Red Hematite or Spathic Ore derivatives (Spathose Yellow, Red, Purple, Brown, Green, and Black); Light Oxide, Purple Oxide, various shades, Crocus; Indian Red, various shades; Colcothar, Turkey Red, Venetian Red, Silicated Oxide of Iron, Ochres, (Yellow, Red and Black); Siennas, Umbers; Vandyke Brown.

CHAP II.—Chemical Colours —Yellows.—Pure Chromes, all shades; Common and Reduced Chromes, Imperial Yellow; Naples Yellow, Cologne Yellow; Mars Yellow and derivatives, Orange, Red and Violet, Golden Ochre, Cadmium Yellow, Strontia Yellow, Zinc Chrome, Barium Chrome; Lithographers' Chromes, all shades, Dutch Pink, English Pink

CHAP III.—Chemical Colours (*continued*) —Blues —Bronze Blue; Chinese Blue, Prussian Blue, genuine and reduced, Soluble Prussian Blue, Brunswick Blue, Celestial Blue; Azure Blue, Pulp Blues; Damp and Paste Blues; Ultramarine and Lime Blues, Oriental Blue; Imitation Herondale Blue, Indiarubber Blues.

CHAP IV — Chemical Colours (*continued*) — Greens — Brunswick Greens, all shades, No. 1 and Reduced Brunswick Greens, Royal Greens, Emerald-tinted Brunswick Greens, Albert and Victoria Greens, Bronze Greens; Quaker Greens, Olive and Sage Greens, Various Mixed Greens, Chrome Greens, Genuine Emerald Green, Reduced Emerald Greens, Imitation Emerald Green, Mineral Green, Green Verditer

CHAP. V —Chemical Colours (*continued*) —Reds.—Red Lead, Reduced Red Lead, Imitation Red Lead, Scarlet Chrome, Derby Red, Persian Red, Chinese Red, Carminettes or Imitation Carmine, Vermilionette or Imitation Vermilion; Permanent Reds, Special Scarlets, Victoria Red, Royal Red, Signal Red; Tuscan Red, Indian Red

CHAP VI —Lake Pigments.—Madder Lakes, Cochineal Lakes, Carmine Lakes; Crimson, Scarlet, Carnation and other Lakes, Alizarine Lakes; Aniline Lakes, all shades, Eosine Lakes, Wood Lakes, various, Cheap Lakes.

CHAP VII —White and Black Pigments —White Lead, Dutch or Slack process and Precipitation Process; Sulphate of Lead, Oxide of Zinc; Sulphide of Zinc, Sulphate of Barium and Barytes, Whiting and Paris White, Sulphate of Lime, Gypsum or Terra Alba, China Clay, Ivory and Bone Black; Vegetable and Lamp Black; Carbon or Gas Black, Mineral Black; Blue Black.

CHAP VIII —Various Dry Colours and Sundries.—Bronze Powders, Gold, Silver and Red; Filling-up Powders, Coach Makers' Colours; Stains and Dyes, French and other Dryers, Distempers and Distemper Powders; Encaustic Powders; American Kalsomine and Calcarium

PART II.—Oils and Mediums.

CHAP IX.—Boiled Linseed Oil, Fire Boiled, Steam Boiled, Single Boiled, Double Boiled; Driers for Oil-boiling; Resinate Driers; Raw Paint Oils; Tests and Properties of Linseed Oil, Reduced Boiled Oils.

CHAP. X.—Pale Boiled Linseed Oils; Various Driers used in Pale Boiled Oils; Cheapeners for Pale Boiled Oils

CHAP XI —Oil for Special Purposes, etc.—Zinc Drying Oil, Quick Drying Oils, Paint Oils for Export; Paint Cheapening Oils; Elastic or Non-cracking Oils; Tarpaulin or Canvas Oils; Linseed Oil Substitutes; Drying Resin Oil; Debloomed Resin Oil; Gloss Oil.

CHAP. XII.—Turpentine and Turpentine Substitutes.—Reduced Turpentines; Deodorised Benzine Cheapeners for Turpentine; Deodoriser for Alcohol.

PART III.—Stiff Paints and Paste Colours.

CHAP. XIII.—White Paints, Lead, Zinc, &c., Special Export qualities for all markets.

CHAP. XIV.—Paints whose Base is Oxide of Iron.—Red Oxide, Spanish Brown and Venetian Red Paints, Turkey Red Paints; Indian Red Paints, Purple Oxide and Purple Brown Paints, Prepared Spathic Ore Paints, Magnetic Oxide of Iron (Iron Minium) Paints.

CHAP. XV.—Sienna (Raw and Burnt) Paints, Umber (Raw and Burnt Turkey) Paints, English Umber Paints, Vandyke Brown Paint.

CHAP. XVI.—Oxford Ochre Paints; Italian Ochre Paints; Stone Ochre Paints; Ochre Paints for Staining, Ochre Paints for Body Colours; Yellow and Chrome Paints.

CHAP. XVII.—Black Paints.—Vegetable and Lamp Black Paints; Black Paints for tinting; Black Paints for body colouring; Ivory Black and Carbon Black Paints, Jet Black Paints for Decorators, Cheap Black Paints; Black Oxide Paints; Black Paints containing White Lead.

CHAP. XVIII.—Red Paints.—Royal, Signal, and Post office Red Paints; Chinese and Persian Red Paints, Maroon Paint, Paste Lakes.

CHAP. XIX.—Blue Paints.—Prussian or Chinese Blue Paints, Brunswick Blue Paint; Celestial Blue Paint; Azure Blue Paint; Ultramarine and Lime Blue Paints.

CHAP. XX.—Green Paints.—Genuine and reduced Emerald Green Paints; Brunswick Green Paints; Bronze Green Paints, Quaker Green Paints, Bright and Emerald-tinted Green Paints, Olive Green Paint, Imperial Green; Green Silicate Paint, Mineral Green.

CHAP. XXI.—Tinted Paints.—Priming Paint, Buff, Stone, Slate, Lead, Mast and Teak Colour Paints.

CHAP. XXII.—Colours ground in Turpentine, Colours ground in water.

CHAP. XXIII.—Stiff Paints for Special Purposes.—Artists' Tube Colours, Silicate Paints, Carbon Paper Tracing Paints; Ships' Funnel Paints, American Paints for Coaches, Cabs and Engines, Paints for Ironwork and Machinery; Weatherproof Paints, Enamel Paints; Disinfecting Paints, Luminous Paints; Ferrodor Paints; Water Paints: Encaustic and Washable Distemper Paints.

CHAP. XXIV.—Putty (all qualities).

CHAP. XXV.—Driers.—Patent Driers; Manganese Driers; Lead or Litharge Driers; Pale and Dark Driers, Zinc Driers; Driers Solutions.

PART IV.—Thinned Paints, Specialities, and Sundries.

CHAP. XXVI.—Ready-mixed Paints (all shades) for Decorators', Painters', or Domestic Use; Enamel Paints; Methods of Cheapening Ready-mixed Paints; Paint Extender or Cheapener; Flatted or Dead Surface Oil Paints.

CHAP. XXVII.—Thinned Paints for Special Purposes.—Anti-corrosive Paints, Funnel Paints; Engine Paints; Boiler Paints; Bridge Paints; Tin Can Paints; Ironwork Blacks; Heat Indicating Paints, Enamel Paints, Gasometer Paints; Floor Paints, Flexible or Canvas Paints; Linoleum or Oil Cloth Paints, Tarpaulin Paints; Trunk Paints; American Roof Paints; Elastic Waterproof Paints.

CHAP. XXVIII.—Silicate, Water and Distemper Paints.—Fireproof Paints, Washable Paints; Silicate of Soda Water Paint; Binders for Water Paints; Emulsion Waterproof Paints; Kalsomine or Distemper Paints, Fire Resisting Kalsomine; Fire Resisting Paint.

CHAP. XXIX.—Sundry Special Paints.—Mica Lustre Paint; Asbestos Paints; Luminous Paints; Metallic Aluminium Paints (tinted).

CHAP. XXX.—Sundry Compositions, &c., for Special Purposes.—Fireproofing Compo.; Damp-proof Compo. and Petrifying Liquid; Water glass; Lime Killer; Transparent Damp-proof Liquid for Walls: Paint Remover; Wood Fillers, Paste and Liquid; Iron Filler; Rivet-head Compo, Cements for Boilers, Steam Pipes, Roofs, &c.

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